

THESIS

HYDRAULIC CHARACTERISTICS OF FEEDLOT MANURE IN AN ANAEROBIC LEACHATE BED REACTOR

Submitted by

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ABSTRACT

HYDRAULIC CHARACTERISTICS OF FEEDLOT MANURE IN AN ANAEROBIC LEACHATE BED REACTOR

Concentrated animal feeding operations (CAFOs) use the practice of optimizing space for the raising of livestock. By implementing space-saving techniques, these operations end up with large quantities of waste on small parcels of land. One way to utilize the waste is to integrate an anaerobic digester into the waste management approach of a CAFO. Anaerobic digesters efficiently break down waste while creating an energy source.

In the Midwestern United States, water is in abundance, and therefore can be added to continuously stirred reactors or other anaerobic digestion technologies. In semi-arid climates, such as in Colorado, water is a treasured commodity. A new technology is being investigated to limit the need for water addition in anaerobic digestion. Water is trickled through a column of manure creating leachate. Leachate is continuously recycled through the leachate bed reactor, and then flows to a composting tank and ultimately to a high rate anaerobic digester where methane is produced. This method has been used in manure, food and landfill applications. In many cases, clogging occurs either initially or after some digestion has occurred, and the pore space decreases. The objectives of this research were to gain a better understanding of what additives will aid in better flow through manure and to develop a method to characterize hydraulic flow through a column of manure.

Intrinsic permeability (k) was measured with respect to compressed air as the permeant fluid on a homogenized sample of feedlot manure. The impact of compression, bulking agents (straw and wood chips), sieving out small fragments, and dispersion media were compared on the basis of the measured k . Applied force, or compression, had the greatest impact on k because the tested manure was greater than 30% air by volume. Straw showed the greatest increase in k of feedlot manure compared to wood chips and particle sieving.

After determining which substrate combination would be best suited for liquid flow, experiments based on the mean residence time (\bar{t}) were set up. No substrate was added in these experiments, because the behavior of the manure was uncertain. Water with an oxidation reduction potential (ORP) of less than -500 mV was used to mimic anaerobic conditions. Three replicate columns were constructed and operated for six weeks. Three tracer tests, each with a hydraulic loading rate of 0.88 m/d, using sodium bromide (NaBr) as the tracer salt were conducted for three replicate columns. Variability in effluent concentrations and flows was observed in the columns, which was expected due to variability in packing within the column.

The average \bar{t} was approximately 6 hours. Also, the majority of the tracer (60% of the output) leached from the columns in less than one pore volume. Thus, the columns likely experienced preferential flow in that large pulses of water would exit at random times and the majority of the tracer exited the column in less than one pore volume. Tailing of residence time distribution curves and inability to recover all injected salt indicates the likelihood of dead zones within reactors. A ratio (further referred to as ratio R) of the superficial velocity to the hydraulic loading rate was calculated. The ratio R was greater than one for every tracer test indicating that water flows through the column slowly. This indicates retardation through the column consistent with the observation of tailing in residence time distribution curves, also indicating the presence of dead zones. The free drain volume was a small fraction of the pore volume in the total column. The fact that so few pore volumes exited the column indicates severe retardation within the column that can be attributed to dead zones. Each tracer test also showed that very few pore volumes exited the column. This could possibly indicate a retardation in flow.

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1.0 INTRODUCTION

1.1 Research Justification

Concentrated animal feeding operations (CAFOs) only account for 5% of all animal feeding operations (Ag Census, 2007), but contain 50% of all the animals in feeding operations (Gurian-Sherman, 2008). The livestock industry is a profitable industry, but results in generation of a large amount of waste (Gurian-Sherman, 2008). Unlike human waste generated in urban areas, there are limited financial resources available to support on-farm waste management (Gurian-Sherman, 2008). The high organic content of the waste renders it challenging to efficiently treat using conventional aerobic treatment approaches such as continuously stirred or plug flow reactors (Demirer and Chen, 2005).

Anaerobic digestion (AD) is a process to stabilize high-strength wastes while creating an energy source (Demirer and Chen, 2005). Several studies have been performed to evaluate the economic feasibility of anaerobic digestion of agricultural wastes for energy production (Andersson et al. 2002, Demirer et al. 2005, Demirer and Chen 2008, Hills et al. 1981). The key factors which impact economic feasibility include transport of the methane gas, efficiency of the methane conversion to electricity, and the current cost of electricity in the area of study. The data show that digestion is economic in some locations, and not in others (Svensson, 2007).

Anaerobic digestion requires four processes (Demirer and Chen, 2005). The first is hydrolysis (liquefying solids to soluble monomers) followed by two acid stages, and finally methanogenesis. Hydrolysis is the slowest of the four stages in anaerobic digestion due to the complexity of manure (Myint et al. 2009). A common procedure is to place the manure in a moist environment to enhance the remaining three stages of digestion (Demirer and Chen, 2008).

Many CAFO operations are located in semi-arid climates due to relatively large land requirements (Miller and Berry, 2005). These regions tend to have fewer people and more open space. In wetter climates, and limited dairy operations in Colorado, CAFO operations use water to flush or

scrape the concrete floors of the operation. Typical management practices for CAFOs in Colorado include scraping with a machine, leaving the livestock in outdoor corrals where waste is collected periodically and placed in large piles, or flushing pens periodically with water (Sharvelle et al., 2011). In arid regions, water is too valuable of a resource to use on waste treatment.

To avoid using water in digestion, a new technology is being developed in which water trickles through manure producing a leachate. This leachate then is recycled through the process, feeding the system with an already-soluble food source (see Figure 1.1). Diemirer and Chen (2008) have had some success with this process. However, they also report an issue related to the water not flowing through manure. For leachate-reactor AD systems to be successful, hydraulic flow through manure must be better understood and optimized.

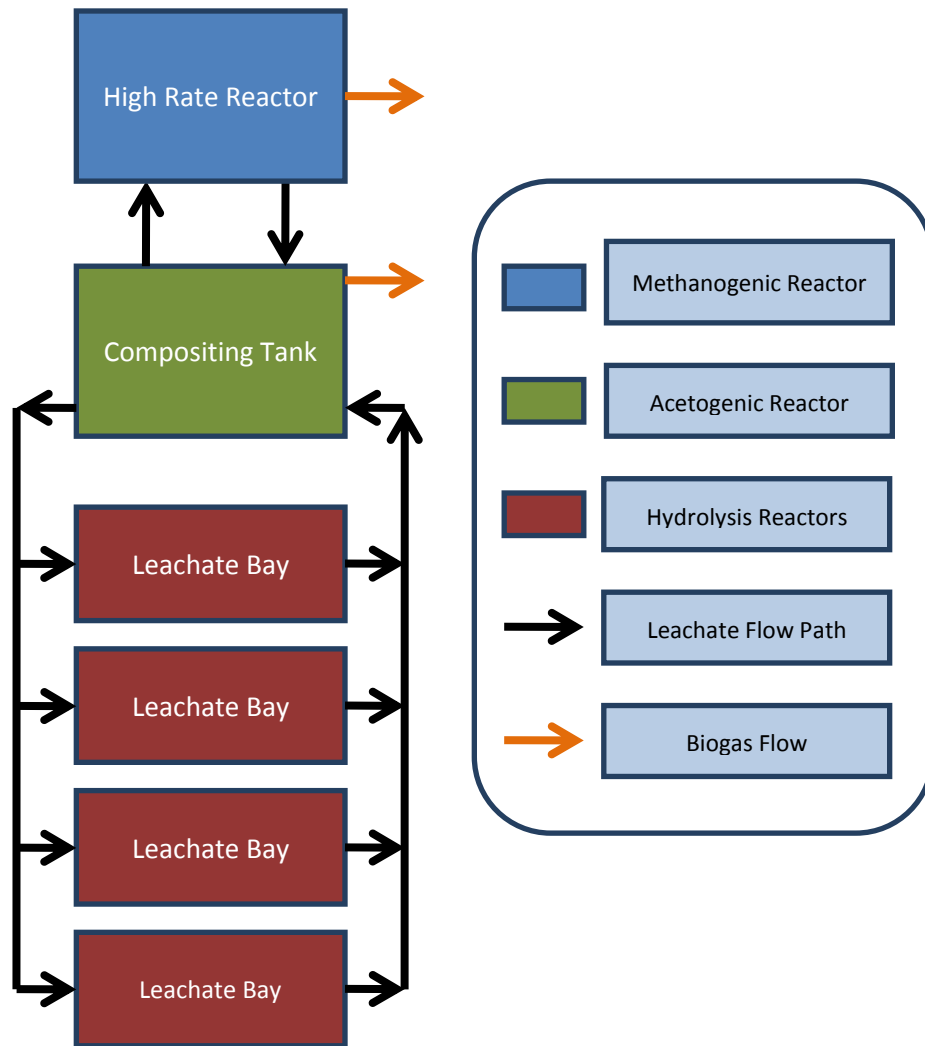


Figure 1.1. Proposed Anaerobic Digestion Process (Loetscher 2012)

1.2 Research Objective

The objective of this research was to identify the best methods to create improved hydraulic characteristics in a leachate reactor containing dry lot collected manure. Several methods were utilized including the addition of bulking agents as well as liquid distribution media. Intrinsic permeability (k) was utilized to gain an initial understanding of the factors that greatly impact flow through dry lot collected manure. In addition, tracer tests were conducted on operating leachate-reactor columns to characterize the hydraulics.

In porous media, the hydraulic conductivity (K) is determined to better understand flow through the media. This determination typically requires completely saturating the sample with water. With manure, maintaining hydraulic flow under complete saturated conditions generally is not possible, since the manure becomes a slurry, which tends to prevent water passage. That is when the manure becomes a slurry, the manure is viscous and water ponds on top due to lack of pore space, characteristics that are similar to those of fresh manure. To bypass the need for complete saturation while still gathering data on multiple substrates, testing was conducted to determine based on air as the permeant fluid.

2.0 BACKGROUND AND LITERATURE REVIEW

2.1 Anaerobic Digestion

Feedlots are in abundance in the Midwestern and Western United States, and produce large amounts of waste each year (Gurian-Sherman, 2008). An operation has to remove 1.8 metric tons of manure per head of cattle each year from the lot (Sweeten and Reddel 1979). Cows are used for dairy production, which has a similar problem with the elimination of so much waste (Sharville and Loetscher, 2011). Collection and disposal of this waste can cost between 12 and 20 percent of the total operation budget for a feedlot (Gurian-Sherman, 2008). It is difficult to use the raw waste as fertilizer due to the high pathogen population (Demirer and Chen, 2008). The mountains of waste that are generated in these facilities cause many environmental problems such as odor, dust, greenhouse gas emissions, and water contamination (Miller et al. 2005). Ammonia and sulfur are frequently found in manure and can cause severe odor problems (Mackie et. al 1998). Of note is that manure can be stabilized through anaerobic digestion or composting to have lower pathogen communities.

Many ideas have been introduced to deal with animal waste management. The waste can be treated like municipal waste, by means of filtration, aeration, and nutrient removal. Most animal operations do not have the resources available to go to such great lengths for no financial benefit (Gurian-Sherman, 2008). The most common practice for waste management is composting (De Baere, 1984). Another approach to stabilize manure is to anaerobically digest it. Anaerobic processes take much more time to stabilize waste than the municipal approach to wastewater (aeration), but create an energy source while using minimal amounts of energy to complete the process (Demirer and Chen, 2008).

The anaerobic process has four main components: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Demirer and Chen, 2008). The hydrolysis step involves converting solids to soluble

monomers, and is usually seen as the rate-limiting step in digestion of manure (Chen et al. 2008) due to the fact that the food for cows is tough to break down to begin with, what they cannot digest is even more difficult to hydrolyze. The acidogenesis and acetogenesis steps create volatile fatty acids, but also produce carbon dioxide (CO_2). Methanogens utilize the CO_2 as an electron acceptor to produce methane (CH_4). Each process has a negative feedback loop meaning that the byproducts of each step are toxic to organisms which perform the same function (Chen et al., 2008). If one step in the process is limited, the other three will also be limited. Hydrolysis is considered the rate limiting step in the process when manure is the substrate being utilized because lignin and other challenging compounds to break down are abundant in manure (Chen et al. 2008). To aid in faster hydrolysis rates, water is often added to dry substrates to encourage already soluble components to separate from solids and become biologically available for the acidogens, acetogens and methanogens (Demirer et al. 2005). Many manure management practices add water to the manure to simplify transport of the material to lagoons.

Anaerobic digestion is beneficial in that it is a net-energy producing method for treating waste, as opposed to aerobic treatment, which is a net-energy consuming approach. The end products of anaerobic digestion are common additives to agricultural areas. These products include humus and other nutrient-rich compounds which can be used in agricultural and park environments.

2.2 Review of Anaerobic Digestion Technologies

Several types of anaerobic digestion exist. The correct technology depends greatly on the substrate (Nallathambi 1997). The most commonly used technology is completely mixed digestion, or a continuously stirred tank reactor (CSTR) (Sharvelle and Loetscher, 2011). In a CSTR, the feed is continuously added to the reactor which has a propeller or other form of mixing agent that does not involve aeration. Typically CSTRs use between 5-10% solids substrates (Sharvelle and Loetscher, 2011). This process is beneficial because there are no stagnant zones and it is relatively easy to implement.

Another continuously-fed digestion process is plug-flow. In the reactor, a plug of waste is added to the process. Plug-flow reactors are generally used with materials having 11-14% solids (Sharvelle and Loetscher, 2011). These conventional methods are not appropriate for the high solids content in the waste tested (Demirer and Chen, 2005).

Other commonly used technologies are high rate reactors such as upflow sludge blanket (3-7% solids) and fixed film reactors (less than 3% solids; Sharvelle and Loetscher 2011). In the upflow sludge blanket a layer of sludge is allowed to grow at the bottom of the reactor. Waste is pushed upwards through the system where anaerobes eat away at the “blanket”. In attached growth systems, the anaerobes grow on a media which allows them more stability due to the fact that they do not exit the reactor unless they detach from the biofilm.

Two less conventional methods of digestion are two-stage and dry digestion (Demirer and Chen, 2008). Two-stage digestion is recommended for agricultural substrates. In this process, hydrolysis and acid production occur in the first stage and methanogenesis happens in the second. The process is usually done in batches so as to avoid overloading the system. More gas is produced using this system due to the inhibition of byproducts. Normally byproducts adversely impact the following step in digestion (the byproducts of acetogenesis negatively impact methanogenesis). Two-stage digestion requires a large area to accommodate both reactors. It is also limited by the hydrolysis rates because this is the first step that jump starts the second reactor. Dry anaerobic fermentation is useful for substrates that have a large percentage of solids (over 25%), such as manure in an arid environment (Demirer and Chen, 2008). Dry fermentation takes a very long time for substrates that have slow hydrolysis rates. With no aid from additional water, this process can require long reaction periods and thus very large reactors.

2.3 Manure Management Practices in the Arid West

In Colorado, the dairy industry collects manure using different methods compared to other parts of the U.S. (Sharvelle and Loetscher, 2011). In eastern and mid-western areas, water is in abundance and dairies can flush barns to eliminate waste. In arid climates, dry lots are applied where manure is scraped (Sharvelle and Loetscher, 2011). Manure on a dry lot excreted at 10-14% solids can lose moisture in an arid climate resulting in more than 90% solids at the time of collection. As mentioned, a waste containing over 25% solids is best suited for a two-stage process. With a solids content as high as 90%, water is needed to enable the hydrolysis process (Sharvelle and Loetscher, 2011).

A new technology is being developed to anaerobically digest manure by utilizing a multi-stage process, including a leachate bay reactor.

2.4 Proposed Multistage Anaerobic Digestion Technology

Figure 1.1 (Section 1.1) shows the basic schematic of what the proposed multi-stage anaerobic digestion technology includes. The basis of the technology is to take a small amount of water (fresh water or wastewater) and apply it in an even distribution manifold at the top. The water drips into a column of manure. This will allow all the soluble organics to be leached from the waste. At the bottom of the column will be a collection system for leachate.

Following the leachate bay reactor is a compositing reactor or a leachate storage tank. The final stage is a high rate anaerobic digestion reactor, such as a fixed film or upflow sludge blanket reactor. Leachate is not only recycled through the leachate bay reactors, but also through the compositing tank. The separation of the hydrolyzed leachate from the solid manure allows acido/acetogenesis to occur. In the high rate reactor, microorganisms are retained in the system resulting in decreased hydraulic retention time and increased methane production.

Some have used a similar approach to the multistage high solids reactor in landfills (Reinhart 1996, ten Brummeler 2000) and manure (Demirer et al. 2008). Many systems in Europe use a similar

technology for food waste. GICON® created a technology called Harvest that uses a two-stage technique to create biogas in which the first stage uses “hydrolysis percolators” to create soluble products followed by anaerobic digestion chambers for gas production. CH2M Hill invented a similar technology called SWANA Evergreen. In this process, the food waste is co-digested with wastewater sludge in a two-stage system using percolation in the first stage as well. More technologies include the BEKON process, BioFirm™ which includes co-digestion of many substrates and recycled leachate, and the Kompoferm® process.

A common problem persists in all leachate bed based systems; after time the system does not pass water (Svensson et al. 2007, Andersson et al. 2002, and Beaven et al. 2005). This phenomenon has commonly been attributed to biological growth clogging liquid pathways. However, clogging can happen instantaneously with manure due to the formation of a slurry. A common solution is to add bulking agents. Some have tried adding pistachio shells (Myint and Nirmalakhandan, 2009). Others have attempted adding straw (Svensson et al. 2007 and Andersson and Bjornsson 2002). The purpose of these bulking agents is to try and keep the manure from becoming a slurry by opening channels for water to flow.

Clogging not only stops the production of leachate, it also decreases the organic content of leachate collected from the system. Mass transfer is limited when flow is hindered through a system (Myint et al., 2009). This means that the flow of water aiding in the hydrolysis process is not sustained, and the production of soluble monomers will remain limited. The cascading impact is that the methanogens in the second stage of the system are limited. Hydraulics directly relate to the hydrolysis efficiency of the system. If more soluble compounds are extracted and recycled, then the extent of hydrolysis will increase. The understanding of dead zones and preferential flow in a system allows for a better understanding of leachable materials. If the substrate has many dead zones, hydrolysis will be limited.

2.5 Background on Characterization of Liquid Flow through Porous Material

Preferential flow was first used to describe the seepage of liquid through fissured rocks (Barenblatt et al. 1960). Gerke (2006) defines “preferential flow comprises all phenomena where water and solutes move along certain pathways, while bypassing other volume fractions of the porous soil matrix.” That is to say preferential flow describes the flow through macropores in soil that speed transport. Coppola et al. (2009) states there are four components of preferential flow: 1. preferential flow occurs in real macropores, 2. it also occurs in inter-aggregate pores, 3. can be affected by fingering along the wetting front, and 4. can be due to special irregularities in the soil or “temporal dynamics” in wettability. All four of these phenomena may occur in leachate bed based AD systems, including the high solids process described in Section 2.4. The heterogeneity of the matrix and the inter-particle pores create a non-uniform flow through the porous media. The wetting front is also non-uniform due to the fact that water does not move through the entire substrate at a uniform rate. That is some pockets move water through faster than others, creating preferential flow in certain areas along the wetting front. Preferential flow in intra-particle relationships has been observed in landfill leachate systems as well (Oni 2009).

Understanding preferential flow is difficult in manure. As discussed, it is impossible to completely saturate manure while maintaining hydraulic flow, and measuring suction along the wetting front of such a heterogeneous material is nearly impossible. There is no way to ensure the wetting front is measured along the entire matrix. However, tracer tests give some indication of the matrix inside a medium.

Tracer tests have been used for many applications including soil, landfill, and biotrickling filter systems. There are several approaches to tracer tests. The purpose is to inject a foreign substance into the substrate and see how long it takes for the entire injected mass to exit the system. Dyes and/or soluble salts are commonly used as the injection media. In landfills, dyes have been found to have little

recovery from the system (2%) due to sorption (Oni 2009). Salts, usually a bromide or chloride compound, are more commonly used for substrates with high sorption affinity (Oni 2009). Bromide is most commonly used since it is not as common as chloride naturally in soil or manure.

Most disciplines describe a breakthrough curve (BTC) as the curve of the concentration of injected tracer exiting the substrate over time. For many tracer systems, this discharge flows at a constant rate. What is most important with non-uniform soils is the cumulative mass that exits the system over time (Shackelford 1995).

Many have documented solutions and pore matrix models for landfills and soils (Andersson et al. 2002, Beaven et al. 2005, Butter et al. 1989, ten Brummeler 2000, Oni 2009, Phelan et al. 1996, Reinhart 1996, Rosqvist and Destouni 2006, Svensson et al. 2007). Each has a different approach to describing such a complex matrix that is not easily visible. From all of these documents, one message is clear; modeling a 3-dimensional matrix for porous media is challenging. The inability to completely saturate the media and an ever-changing hydraulic gradient make it impossible to get a snapshot in time that represents the entire matrix (Wu et al., 2012).

2.6 Hydraulic Characterization of Liquid Flow through Waste Material

In soils, permeability values are measured by completely saturating a sample and adjusting the hydraulic gradient to move water through. This approach is not possible in landfill or manure substrates because they degrade over time, and manure creates impermeable slurries when it is completely saturated. However, some soils concepts can be applied to landfill systems that operate similarly to manure systems.

Some researchers have evaluated degradation of landfill material over time. This not only decreases the hydraulic conductivity, but also the strength of the waste (Reddy et al. 2009 and Vilar and Carvalho 2004). The composition of the material impacts the degradation (Reddy et al. 2011), and smaller fragments tend to degrade faster than larger fragments.

Landfill systems can experience rapid settling due to this degradation and compression of void spaces (Olivier et al. 2006, and Reddy et al. 2011). This rapid settlement can lead to clogging. Some have reviewed methods of deterring clogging in landfill systems (Fleming and Cullimore 1999, Valencia et al. 2009, and Yu and Rowe 2011). Valencia et al. (2009) studied various configurations of porous layers in the landfill: gravel on the bottom, mixed within the system, and no gravel at all. It was found that the system with gravel mixed in was the most successful. The other systems clogged within a year. Olivier et al. (2006) discovered that particulates and microbes were the main reason for clogging in the gravel at the bottom of the system. Yan Yu and Kerry Rowe (2011) used sand as the drainage layer, which was less successful than only gravel due to the lack of pore space. However, the system did show an increase in permeability with increasing amounts of sand. Like landfills, manure degrades with time, creating smaller pores for flow. Manure also contains a lot of air and can settle rapidly, decreasing the pore space. The addition of a bulking agent would be necessary in manure systems for the same reason it is necessary in landfill systems.

2.7 Addition of Bulking Materials to Waste Products

Research in Sweden (Andersson et al. 2002; Svensson et al. 2007), is looking toward renewable energy from waste products due to increasing oil prices and attempting to lower greenhouse gas emissions. Digestion of agricultural waste has not been as heavily analyzed as landfill digestion. This group of scientists (Andersson et al. 2002; Svensson et al. 2007) is investigating the addition of bulking agents such as straw in landfills to enhance digestion. The data suggest that straw is an affordable and beneficial addition to anaerobic digestion (Andersson et al. 2002; Svensson et al. 2007). Straw increases gas production by providing an extra carbon source that is more biologically available than manure. However, the straw also serves as a “particulate and biofilm filter” (Svensson et al. 2007). In these experiments, straw was used to aid in anaerobic digestion, and an increase in permeability was found as an additional benefit.

Several studies have shown an increase in methane production of manure with straw addition in stratified bed reactors (Hills et al. 1981, Møller et al. 2004, Wujcik and Jewell 1980). However, little is known about how it affects the permeability of leachate bed reactors packed with manure. Based on the data from Sweden (Andersson et al. 2002; Svensson et al. 2007), straw appears to be more beneficial than harmful. However, not all agricultural areas have access to straw. Myint et al. (2009) added pistachio-half-shells to increase the porosity of their digestion process. Data suggest that the addition of bulking agents is necessary to prevent clogging in anaerobic leachate bed reactors (Myint et al. 2009, Andersson et al. 2002, Svensson et al. 2007). Adding a bulking agent that also increases degradation of waste is most beneficial to the system.

In general, research shows that clogging does occur in landfill systems and that the addition of straw was beneficial. Straw aids not only in better gas production but also in maintaining pore space over time. Straw seems to be a reasonable bulking agent that degrades well. As mentioned previously, gravel was mixed in a landfill system (Valencia et al. 2009). This works well, but requires the addition of a non-degradable bulking agent.

2.8 Summary

The literature shows that preferential flow and clogging are prevalent in food waste, municipal solid waste, and manure. Some have tried the addition of other substrates such as pistachio shells or straw to overcome clogging. Others have tried adding gravel to the effluent to maintain pore space in the effluent. Two-stage AD systems must maintain a steady flow to be effective. Hydrolysis depends on the transfer of fluid through the manure. If the hydrolysis step is limited, every other step is also limited. The flow through a column is the basis of the high solids digester. Little work has been done to characterize hydraulic flow through manure in a leachate bed reactor, particularly since researchers have not determined approaches to sustain liquid flow through manure long enough to enable complete hydrolysis.

3.0 ESTIMATION OF INTRINSIC PERMEABILITY IN MANURE

3.1 Introduction

As explained in Section 2.5, traditional methods for characterizing hydraulic properties in soil are not appropriate for analysis of manure because flow cannot be maintained in completely saturated conditions. Therefore, understanding the hydraulic characteristics, specifically the hydraulic conductivity, is challenging. Since water could not be used to saturate manure samples, compressed air was used as the saturating medium and as the permeant fluid. With a completely air saturated sample, the intrinsic permeability, k , could be determined. The resulting comparison of values of k was useful in terms of providing a better understanding of which substrate combinations should be used in future experiments when liquids, instead of gases, would be applied to the leachate reactor. Thus, air was passed through a column of manure with varying loading and bulking agents. The value of k was determined, and the various values of k based on different testing conditions were compared to establish trends and provide conclusions of the observed behaviors.

3.2 Methods

3.2.1 Experiment Setups

In the experiment, 299 columns of waste material with varying compression and bulking agents were placed in the testing apparatus. The experiment involved applying compressed air to the top of columns and testing for the pressure differential across the columns under constant flow rate conditions. The columns then were saturated completely with air, and k was determined. For most experiments, three columns were tested simultaneously for analysis of each addition of bulking agent and energy applied. The straw additions were 0%, 0.05%, 0.1%, or 0.2% by weight. Each of the straw weight additions were exposed to 0, 23, or 49 J/m² energy. That is to say three experiments (nine columns in total, three triplicates) were created for each straw addition: one set of columns with 0 J/m²

energy applied, a second set of columns with 23 J/m² energy applied, and a third set of columns with 40 J/m² energy applied. Three more experiments were performed for the 0.05% straw addition: the first set of columns with 0 J/m² energy applied, the second with 23 J/m² energy applied, and a third set with 49 J/m² energy applied and so on. The wood chip additions were, 0%, 0.5%, 1%, or 2% by weight. Similar to the columns packed with manure straw, column containing wood and manure was exposed to 0, 23, or 49 J/m² energy. The manure was also exposed to 0, 12, 23, 37, 49, 116, 233, and 544 J/m² without any bulking agent addition. Sieving separated the manure fragments based on 6 sieves with sizes in order of 6.68 mm, 3.32 mm, 2.38 mm, 0.98 mm, 0.5 mm, 0.295 mm, and the pan. The sample collected in each sieve was only exposed to 49 J/m² energy.

3.2.1.1 Sample Collection and Homogenization

Samples of manure were collected from the JBS Five Rivers Cattle Feeding, LLC in Kersey, CO. The first sample was collected in March of 2011. A representative sample was collected from the top and bottom of each pile of manure as well as from each area of the feedlot. The samples then were sieved to separate smaller and larger fragments using a 19-mm (0.75-in) sieve. The small samples were subdivided into four equal parts by weight. The larger fragments were shredded in a wood chipper (3 horsepower, Harbor and Freight, Fort Collins, CO) and distributed evenly by weight with the smaller fragments. This process is similar to what would be used in the field. Larger fragments would be chopped by an industrial composting chipper to allow for a food source that is more biologically available material. The combined smaller and larger fragments were mixed by hand while being placed in the sample storage buckets. Each of the four even parts of mixed smaller and larger fragments was evenly distributed into 19-L (5-gallon) storage buckets. This stored sample was then loaded into the 299 columns used in this section of the study.

3.2.1.2 Waste Characterization

The waste characterization methods were similar to those used for soil (ASTM D4906 for total solids and ASTM D2369 for volatile solids). The total solids (TS), volatile solids (VS), and specific gravity (G_s) were all determined for the combined smaller and larger fragment samples described in Section 3.2.1.1.

Total solids were determined by evaporating liquid from the sample (5-10 g) in an aluminum pan (76.2-mm (3-in) diameter aluminum pan, VWR) in an oven set at 110°C for 2-h to 6-h (Thelco Lab Oven, Precision). The pan containing the sample was weighed before and after the drying process and the TS was calculated in accordance with the following equation:

$$TS (\%) = \frac{\text{Dry Mass}}{\text{Original Mass}} * 100\% \quad 3.1$$

Volatile solids were determined by removing the sample from of the 110°C oven and placing it in a separate oven set at 550°C for 30 min. The VS was calculated using the following equation:

$$VS (\%) = \frac{\text{Mass After 550}^\circ\text{COven}}{\text{Mass After 110}^\circ\text{COven}} * 100\% \quad 3.2$$

Specific gravity was performed using a 500-mL volumetric flask. The weight of the flask was recorded, and approximately 50 g of sample was added. The weight of the combined flask and sample was recorded. The flask then was filled to 500 mL, and the final weight was recorded. The flask was sealed on top with a stopper and needle through the top. The needle was connected to a vacuum that operated for 36-48 h to eliminate air in the sample. Equation 3.3 shows how specific gravity (G_s) was calculated.

$$G_s = \frac{\text{Mass of Dry Sample}}{\text{Mass of Water Displaced}} \quad 3.3$$

3.2.1.3 Column Construction

The columns used for the majority of the intrinsic permeability testing will hereby be referred to as the “intrinsic permeability testing columns” or IPTC columns are shown in Figure 3.1. These columns

were used for the intrinsic permeability tests because they are easy to load, easy to move from loading zones to the compressed air line, and are proportional to the size of fragments that were added. Other columns were used for liquid flow experiments to be described in Chapter 4. The IPTC were constructed from a 0.58-m (approximately 2-ft) tall, 73.5-mm (3-in) diameter, Schedule 40 (Sch. 40) PVC pipe. At each end of the pipe was a coupling, with a slip fitting at one end and a threaded fitting at the other end. A Sch. 40 PVC cap was threaded into a bushing that was glued to each end. This allowed for easy access from both ends of the column. Both end caps had two holes. In each hole was a 3.2-mm (0.125-in) stainless steel NPT to barb fitting, called a portal. A portal with a ball valve was used for sampling or controlling flow. On the bottom cap only one hole was exposed so the bottom of the column could be assumed to be at atmospheric pressure. The top cap had both holes open; one for compressed air to enter and the other for a pressure differential measurement (see Section 3.2.1.4). At the bottom of the column was a 50-mm² mesh (MSC Supply).

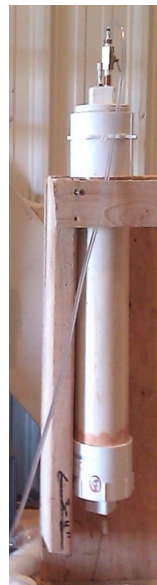


Figure 3.1. Intrinsic Permeability Testing Columns

3.2.1.4 Intrinsic Permeability Testing Apparatus

Figure 3.2 illustrates how compressed air was passed at a constant rate through a reservoir of water with a valve on one of the top portals. The reservoir of water is necessary for the air to build up

pressure. The columns were not constructed to be air tight, so air will just flow through the column without any back pressure, taking much longer to build up the pressure in the column of manure. The second portal on the top cap was connected to a flow meter. The bottom of the column was connected to an overflow bucket approximately 5.8m (approximately 20 ft) above the column of water. This bucket was utilized for overflow from the reservoir. It was mainly used in start-up. The compressed air then flowed to the top of the column filled with manure. The bottom of the manure column was open to the atmosphere. A stopper was placed in the second portal to place a needle attached to a monometer, which measured the pressure at the inflow side of the column which is the same as the pressure drop across the specimen due to the fact that the outflow is exposed to the atmosphere. The gas was allowed to flow for several minutes before a measurement was taken. A needle attached to a monometer was placed in the stopper in the top cap to obtain the pressure differential across the column (see Figure 3.2). It was assumed the bottom of the column was at atmospheric pressure because one of the portals was left open. The reading out of the monometer was the total pressure across the column.

The column was left for a minute to attempt to completely saturate the column with water. Then the needle attached to the monometer was placed in the top portal. The monometer responded to the pressure in the column.

Measuring the pressure differential was challenging using a traditional monometer. The markings on the monometer were not accurate enough, and the pressure differential was too small to move the water a measurable amount. Therefore, a monometer was made at an angle of 20° with 0.635 cm ($\frac{1}{4}$ in) glass tubing. It was filled with deionized water. The pressure would build up in the manure column. This pressure would push the water in the manometer. Pressure was measured by using the conversion of 1 cm of H_2O is 98.1 Pa. The IPTC columns were filled with manure (see the Section 3.2.1.5 for loading techniques).

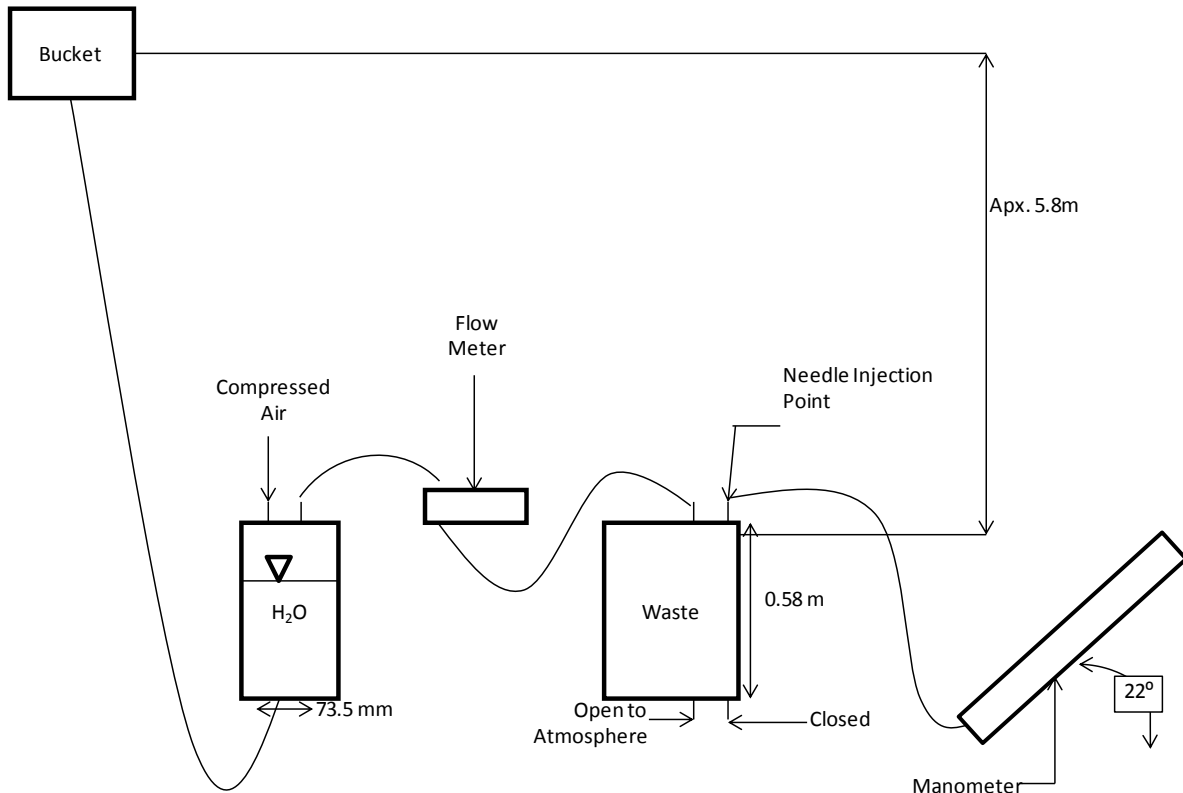


Figure 3.2. Intrinsic Permeability Testing Schematic (Figure Not to Scale)

The calculation of intrinsic permeability was needed after collecting all the data. Equation 3.4 is the definition of intrinsic permeability. It utilizes the conductivity.

$$k = \frac{K \cdot \mu}{\rho \cdot g} \quad 3.4$$

where: k = intrinsic permeability (m^2), K = conductivity (m/s), μ = dynamic viscosity of fluid through the column—air ($1.8741 \text{ Pa} \cdot \text{s}$), ρ = fluid density—air density at standard pressure and temperature (1.18 kg/m^3), g = gravitation constant (9.81 m/s^2).

However, hydraulic conductivity cannot be found in manure because it is challenging to completely saturate the sample. Therefore, Darcy's equation was used to define the conductivity.

$$K = \frac{Q}{i \cdot A} \quad 3.5$$

where: K = conductivity (m/s), Q = flow of fluid (Air) (m^2/s), i = gradient (m), A = cross sectional area of flow (m^2).

Substituting the definition of conductivity (Equation 3.5) into the equation for intrinsic permeability (Equation 3.4), the result is Equation 3.6.

$$k = \frac{Q \cdot \mu}{\rho \cdot g \cdot i \cdot A} \quad 3.6$$

However, only pressure head and elevation head have an impact on this system: there is no velocity and the height difference was considered negligible. The definition of pressure head is Pressure/(density*gravity), which eliminates the existing density*gravity term. Therefore, the gradient becomes dimensionless by dividing the change in pressure by length. Equation 3.7 is the final equation used to estimate k.

The assumptions used in the calculations were based on bulk values in the gaseous phase of the manure.

$$k = \frac{\mu \cdot Q \cdot l}{A \cdot \Delta P} \quad 3.7$$

where: μ = dynamic absolute viscosity of air (1.8741 Pa*s), Q = air flow rate (m^3/s), l = length of manure in column (m), A = cross sectional area of column (m^2), ΔP = change in pressure (Pa). This equation has been derived from the air-flux equation (Corey, 1986).

3.2.1.5 Compression

Experiments were conducted where k was determined for compressed manure. The objective was to evaluate the hydraulic characteristics through manure in a natural state. Also, the columns that will be used in the field (full scale implementation) will be tall, resulting in compression of manure at the bottom of the reactors.

The columns were loaded in lifts. A lift is a term used in soil practices (Standard Proctor Test ASTM D-698 and Modified Proctor Test ASTM D-1557) to describe a set amount of porous material placed in the experimental apparatus to ensure even loading. Each lift was 100 mm for the IPTC columns. That is to say the manure was added in 100-mm increments. The 100-mm increments were measured by placing a meter stick at the bottom of the column, loading the lift, compressing it using a

combination of dynamic and static compaction practices, and placing the meter stick again in the column. The meter stick was placed on top of each compressed lift to measure the next 100 mm. For the intrinsic permeability experimentation apparatus columns, after 100 mm was placed in the column, it was placed in the compression apparatus (Figure 3.3). The bottom had a fitted area made of wood so it would not be on the ground and break during compression. The top of the column was supported by plumber's tape. The columns were compacted using a combination of static and dynamic compaction. The load was dropped on the columns, using the weight of the compacting device similar to a static compactor. However, the wood was inserted between each lift, similar to a dynamic compactor. A wooden circle made from plywood that fit exactly inside the 73.5-mm diameter top was lowered into the column. The circle was mounted on a 12.7-mm (0.5-in) copper pole. Also on the pole was a piece of 19-mm (0.75-in) Sch. 40 PVC with a string set to raise the pipe 127 mm and gym weights making the total weight dropped equal to 1.6 kg. To ensure the energy applied per unit area remained consistent in the IPTC columns, the string height was set to 170 mm and the weight on the PVC pipe was 2.65 kg to maintain the same J/m^2 values used in the intrinsic permeability tests.

One lift (100 mm tall) was loaded in the column and set in the apparatus with the wooden circle inside. The PVC pipe with the weights and string would slide up and down the copper pipe to the height of 127 mm or 170 mm set by the string. The 127 mm height was used to maintain a specified compression (J/m^2) in the IPTC columns and the 170 mm height was used to maintain the same specified compression (J/m^2) in the mean residence time experiments (see Chapter 4). The PVC pipe was then be dropped, letting the only force that acted on it to be gravity. Depending on the experiment, the weights were dropped a specific number of times to change the amount of energy applied (Table 3.1). Equation 3.8 shows how the compression was calculated.

$$C = w * h * n \quad 3.8$$

where: C = compression (J), w = weight dropped (kg), h = height dropped (m), n = total number of drops.

The total number of drops (n) refers to the number of drops per lift multiplied by the number of lifts to give the total number of drops. There was some variability in the number of lifts due to the natural variability of the packing matrix of manure. Therefore, for easier analysis, the number of lifts was rounded to the nearest whole number. Table 3.1 shows the total number of drops, after being rounded to the nearest whole number. The joules of energy applied were then averaged for each value of total number of drops. Figure 3.3 shows the compressing apparatus.

The volume of air that was compressed out of a sample was calculated by taking the difference in the volume from before compression and after compression for one lift. It was assumed that all the volume lost was air due to the fact that no moisture visibly left the column and all solids were contained.

Table 3.1. Number of Drops and Corresponding Joules of Energy Applied to the Column per Unit Area.

Total Number of Drops	Energy (J/m^2)
1	12
2	23
5	49
10	116
20	233
40	543

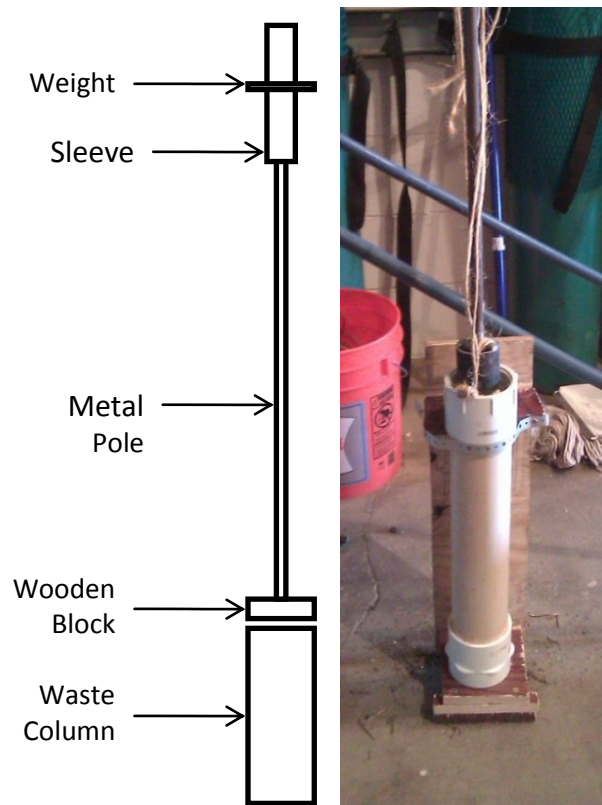


Figure 3.3. Compression Apparatus

3.2.1.6 Bulking Agents, Particle Size Selection, and Liquid Distribution Media

The bulking agents used in this experiment were straw and wood chips. Wood chips and straw were thoroughly mixed into the manure at 0%, 0.5%, 1%, and 2% by weight. This did result in a larger volume of straw being added compared to wood chips. Volume was not used as the basis of measurement because it is difficult to measure volume of such different substrates. Each of the compositions was tested at 0 J/m^2 , 23 J/m^2 , and 49 J/m^2 of applied compression. Straw was added in the same percentages by weight at the same compression values.

In an attempt to understand why manure clogs so rapidly, the smallest fragments ($<4 \text{ mm}$) were removed from the manure. A different set of sieves were used, allowing even smaller fragments to be removed compared to the sorting techniques. The intent was to open more void space for water to pass. Particle distribution was determined by selecting 6 sieve sizes: 6.68 mm, 3.32 mm, 2.38 mm, 0.98

mm, 0.5 mm, 0.295 mm, and the pan. The samples were distributed among the sieves. Each sieve was weighed, and k of each sample was found by placing it in a column and running the intrinsic permeability test.

Media was added to the top of some of the columns (80 of the 299 columns and all of the columns in Chapter 4) to improve liquid distribution throughout. For these experiments, 40 mm gravel was added to the top and bottom of the columns. The gravel was approximately 10 mm in diameter on average.

3.3 Results and Discussion

3.3.1 Waste Characterization

The volume and mass of the solids, water, and air in the homogenized sample were analyzed two weeks after the sample was collected. Figure 3.4 shows the phase diagram for the manure sample. The percentage of solids by volume (V_s) is 38.46%, the percentage of water by volume (V_w) is 29.59%, and the percentage of air by volume (V_a) is 31.95% before compression. The percentage of mass that is solids (M_s) is 61.54%, the percentage of water by mass (M_w) is 38.46%, and the percentage of air by mass (M_a) is assumed to weigh nothing. The specific gravity was determined to be 2.08.

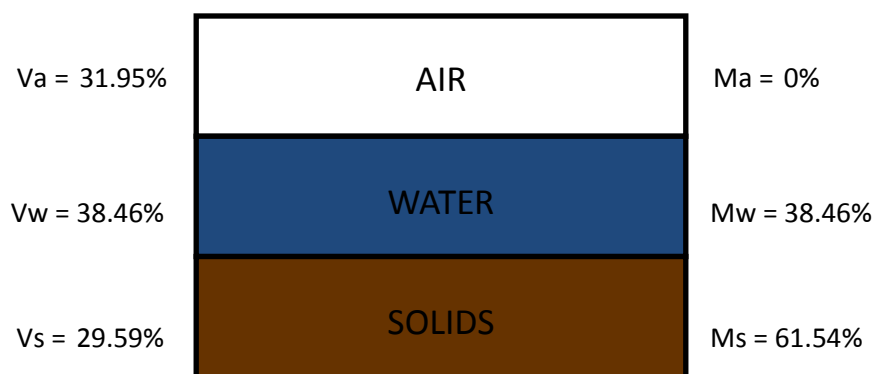


Figure 3.4. Phase Diagram for Manure Samples

The phase diagram shows a large percentage of the manure mass is solids. Therefore, feedlot manure is an excellent candidate for multi-stage digestion. Of note is that the samples were collected

from a feedlot during the spring, when water content is generally higher than summer and fall months. Therefore, manure added to a leachate bed reactor in the field may have even higher solids content than manure added for these experiments. There is no limit to total solids for operation of the proposed multi-stage anaerobic digester. The volume of air is for an uncompressed sample. While preparing the specific gravity experiment, the manure was aerated as it was loaded into the flask. The volume of air value reported in the phase diagram does not correspond to the volume of air that was utilized during the rest of the experiments. The volume of air was decreased by 57.3% when 49 J/m^2 of energy were applied. This further indicates the need for exploration of the impact of compression (Reddy et al. 2009).

3.3.2 Determination of Intrinsic Permeability

3.3.2.1 Compression

The relationship between the amount of energy applied and k found by measuring the pressure differential across a column filled with compressed air was investigated (Figure 3.5). Table 3.2 shows the change in height of the manure in the column based on the energy applied, resulting in a loss of air. Figures 3.5A, 3.5B, and 3.5C demonstrate impacts of compression on intrinsic permeability. The first shows how an increase in energy applied per square meter decreases permeability based on energy applied, bulk density and porosity. Figure 3.5D demonstrates how density increases and porosity decreases with the addition of energy applied to the sample. This concept helps define the elimination of air in samples as they are compressed.

The trend shows a small amount of energy has a large impact on k , but large energy loads have no greater effect than the small loads. Based on this result, it was decided that columns loaded for evaluating liquid flow through manure would have 49 J/m^2 applied energy (see Chapter 4). The energy application saved time by not having to compress samples numerous times, but also is considered representative of a sample in an actual operating reactor. While a large standard deviation was

observed in non-compressed samples, it decreased for samples experiencing more than, or equal to, 49 J/m². The variability in the compression and intrinsic permeability data can be attributed to the air voids. The uncompressed manure contains 31.95% air. This means that the packing configuration will vary drastically every time a column is packed (Higdon and Ford 1996). This explains variability in compacted samples.

Table 3.2. Manure Heights and Volumes Before and After Compression

Energy Applied(J/m ²)	Average Height (cm)		Average Volume (cm ³)	
	Pre Compression	Post Compression	Pre Compression	Post Compression
0	60.0	60.0	456.0	456.0
12	60.0	51.2	456.0	389.2
23	60.0	50.7	456.0	385.5
37	60.0	51.6	456.0	391.8
49	60.0	50.3	456.0	398.5
116	60.0	49.2	456.0	359.1
233	60.0	48.7	456.0	350.4
544	60.0	48.6	456.0	324.5

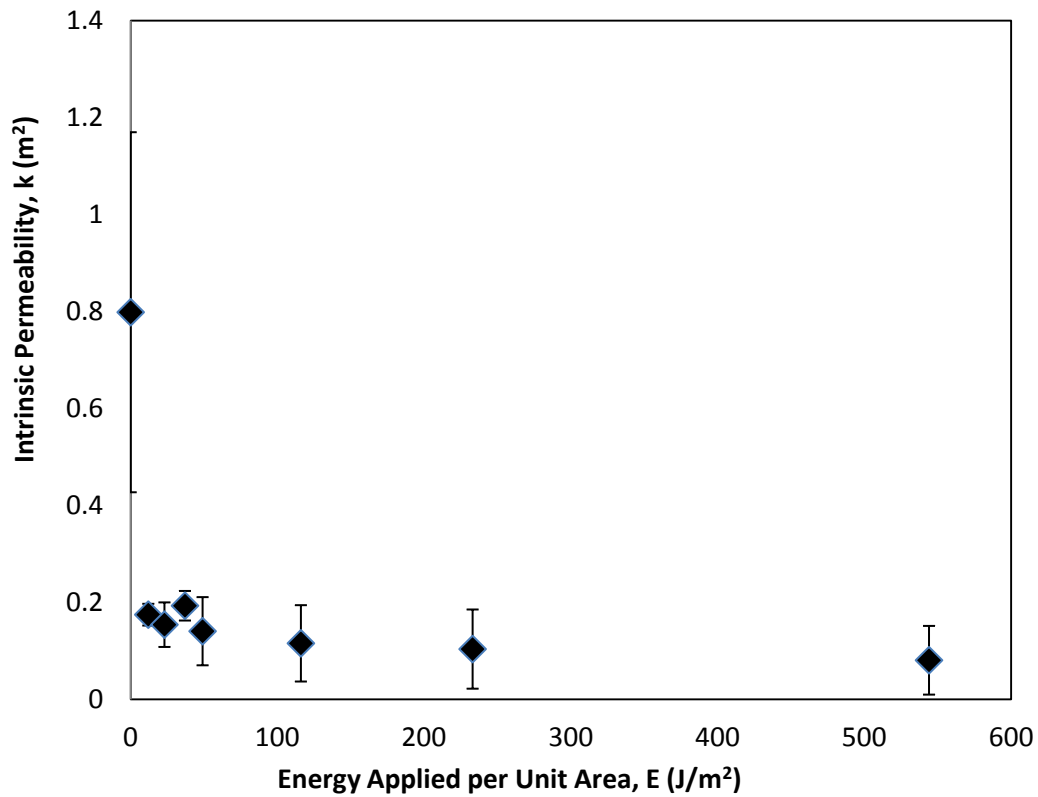


Figure 3.5A. Compression Effects On Manure in Terms of Energy Applied

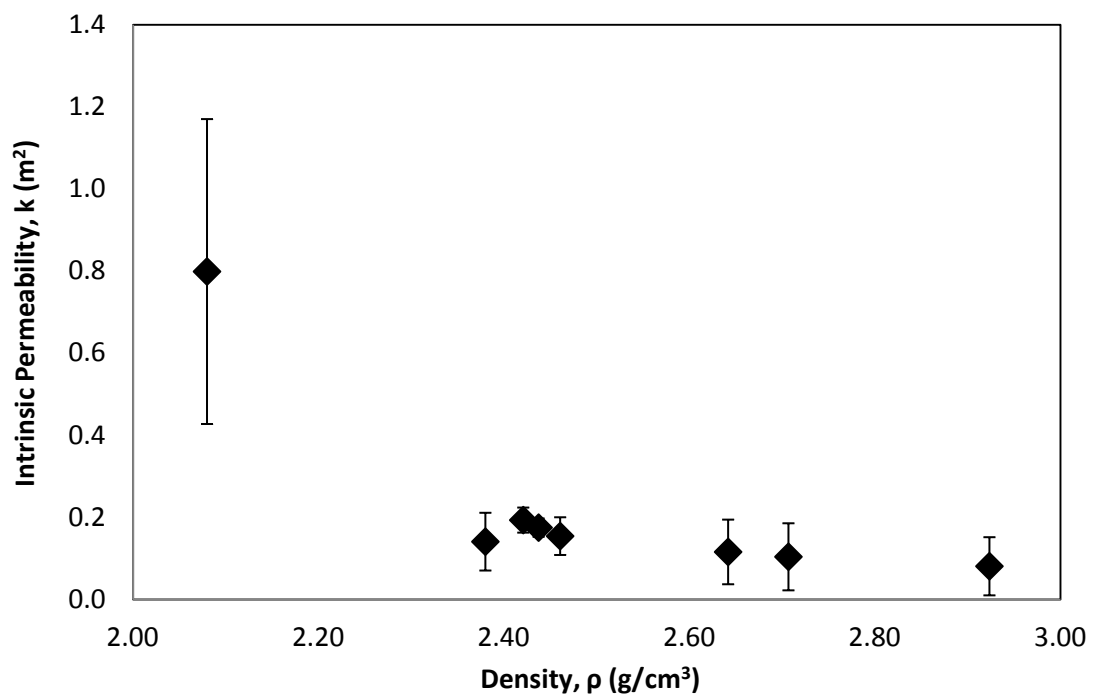


Figure 3.5B. Impact of Bulk Density from Compaction on Intrinsic Permeability

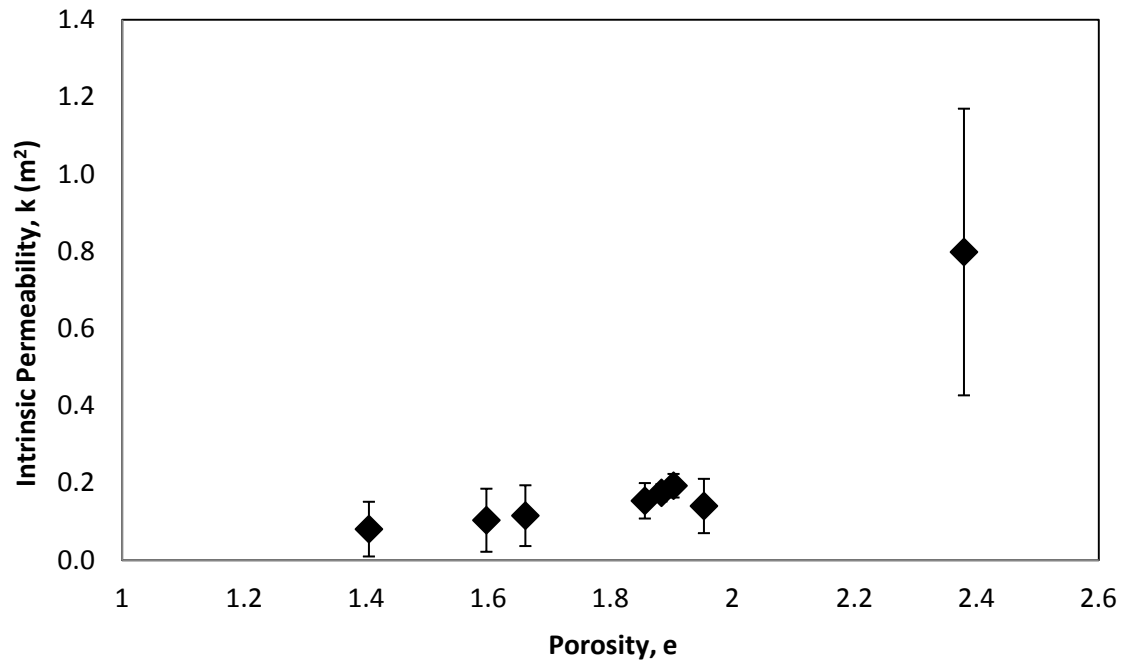


Figure 3.5C. Impact of Porosity on Intrinsic Permeability

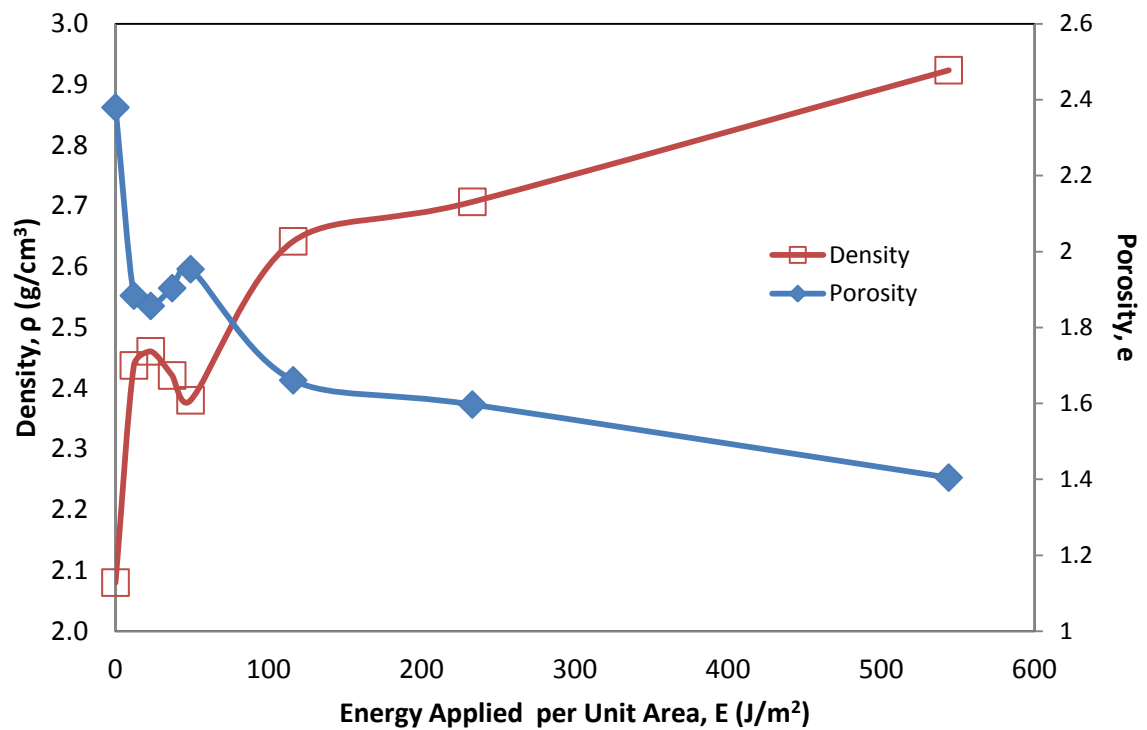


Figure 3.5D. Change In Porosity and Density Compared to Energy Applied per Unit Area

No water exited the columns during compaction, indicating the volume decrease was due entirely due to the reduction of air. The compression data shows that small compression makes a large

impact and a lot of compression is not necessary. Manure can be compacted easily. Once it is compacted, it is difficult to compact it more.

The compressed samples were in a completely dry environment, and experiments were performed within several minutes, leaving no time for biodegradation. In an operational reactor, air voids decrease over time due to biological activity and the creation of more fine fragments (Fleming and Cullimore, 1999). Therefore, compression should help to replicate conditions in a manure column after some time has passed rather than only when the column has just been loaded with fresh manure.

3.3.2.2 Bulking Agents

No upward trends were observed between the amount of wood chips added and k (Figure 3.6). The permeability goes down as the amount of wood chips added increases when no energy is applied. Wood chips are not permeable, and the more that were added, the lower the permeability became. Again, the standard deviation was large for all the samples which were compacted with less than 49 J/m².

Figure 3.7 shows how straw addition impacted k . When no straw was added (0% by weight), compaction had a large impact on intrinsic permeability. However, with 0.1% and 0.2% by weight straw addition, data indicate that straw increased k values for samples experiencing 23 J/m² compression, while increase in straw did not impact k for those samples where 49 J/m² was applied.

The wood chips were chopped to create a more comparable ratio of the size of wood chips to the column, but still did not enhance intrinsic permeability the way straw did. Straw creates channels in the manure that serve as a particulate filter (Svensson et al. 2007). The wood chips are simply too dense to aid in k . However, these experiments were only conducted in the gaseous phase. It is possible wood chips would help initiate bio-degradation or provide structure to a complicated matrix when the liquid flow through a column is used.

Overall, the straw and wood chips did not have a notable impact on k . Ten times the amount of wood chips needed to be added to have a similar impact as straw by percentage by mass. However, research shows that straw aids the digestion process (Andersson et al. 2002, Svensson et al. 2007 Hills et al. 1981, Møller et al. 2004, and Wujcik and Jewell 1980). Further investigations on the benefit of straw and other bulking agents would be beneficial to inform decisions on whether bulking agents should be added. However, these preliminary results indicate that use of straw may be more beneficial than wood chips to improve hydraulic conductivity through manure. Intrinsic permeability is, however, only an indicator of actual hydraulic characteristics.

An attempt was made to explore the relationship between the volume of air in each compressed sample and observed k . However, during the experimental set up, the percentages of straw and wood chips were switched. That is to say, a specific gravity test with 0.1% by weight wood chips and 1% by weight straw was performed. Therefore, the data were not utilized, and the plot was not created.

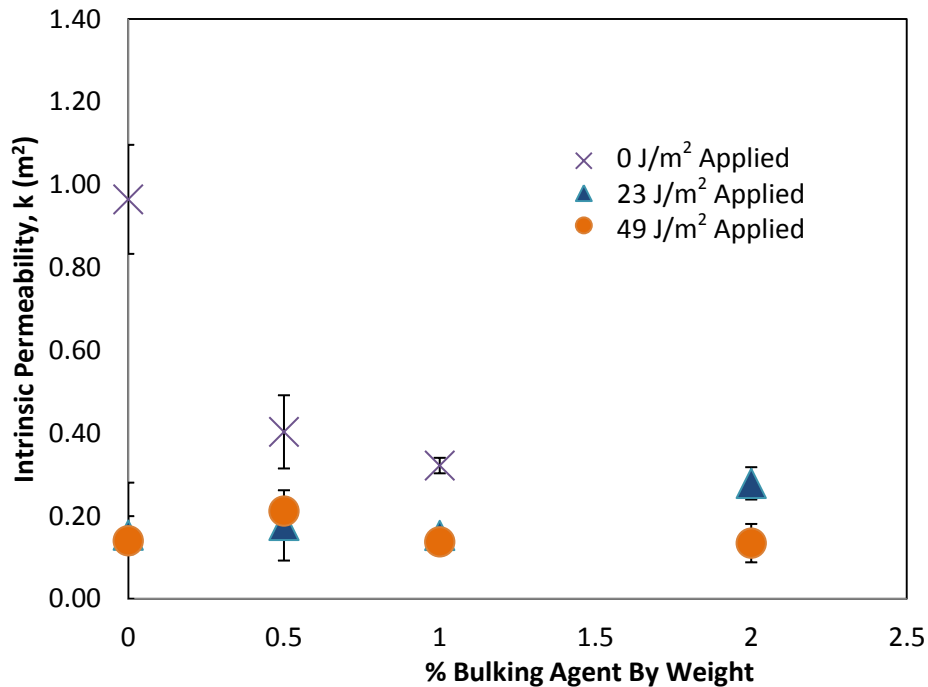


Figure 3.6. Impact of Wood Chip Addition on Permeability

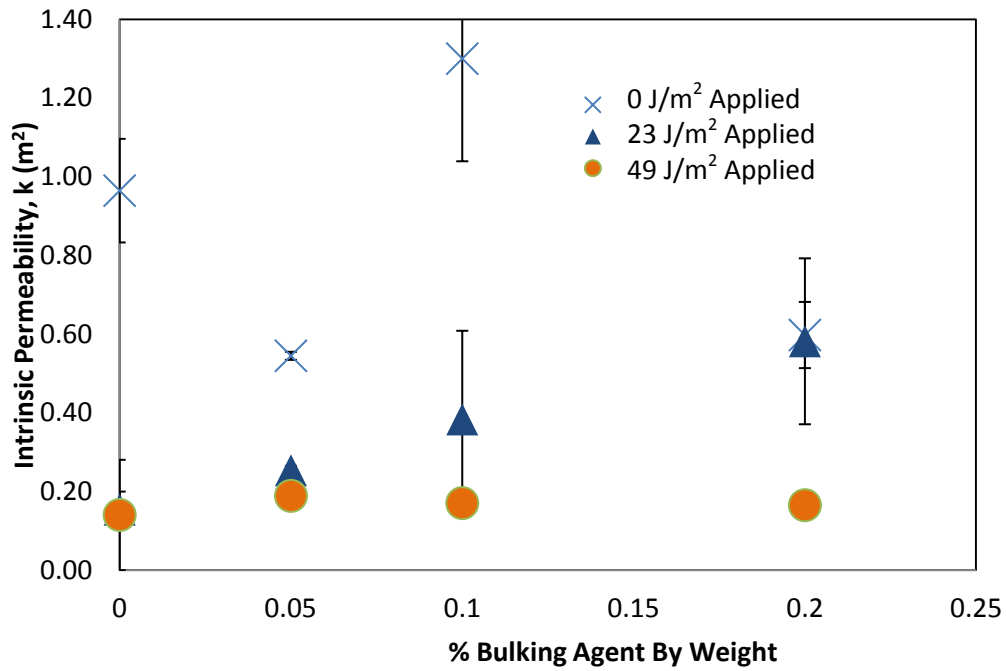


Figure 3.7. Impact of Straw Addition on Permeability

3.3.2.3 Elimination of Small Manure Fragments

Large fragments typically have more void space, and therefore a higher hydraulic conductivity. An attempt was made to determine if hydraulic conductivity of manure could be improved by excluding small manure fragments. Several sieve sizes were used to separate smaller fragments and eliminate them from the columns. Figure 3.8 gives an indication that the elimination of smaller manure fragments to leave only larger fragments in the column increases permeability. A t-test was performed to understand the significance in the data by comparing the intrinsic permeabilities for fragments greater than 4 mm to those smaller than 4 mm. The test used a 2-tail comparison and assumed that the variance was the same for both sets of data. The average value for the permeabilities of sample containing only fragments greater than 4 mm was $0.05 \pm 0.035 \text{ m}^2$ and for the samples containing fragments smaller than 4 mm the average was $0.038 \pm 0.017 \text{ m}^2$. The null hypothesis was that the permeability of samples containing only the fragments 4 mm would have a lower permeability than those containing fragments less than 4 mm. The probability that the null hypothesis will occur was 0.34.

This means that removal of fragments less than 4 mm in diameter does increase permeability, however the difference is not statistically significant at the 95% confidence interval. Of note the larger fragments make up the smallest portion of the sample (Figure 3.8). For example, elimination of fragments less than 6.7 mm resulted in elimination of 68% of the manure on average. An economic analysis would need to be conducted to determine feasibility for such fragment separation. If large fragments were to be separated, an industrial sieve would be recommended for full scale application.

These sieves are commonly used for composting and are typically 12.5-25 mm (0.5-1 in). They are typically used after the compost has been produced. This size is larger than the sieves used in the lab, but would serve a similar purpose. The smallest fragments would be added to compost piles, and the larger fragments could be digested. This could possibly hinder digestion because smaller fragments may be the most accessible to the microorganism community, thus initiating hydrolysis. Of note is that none of these samples were compressed.

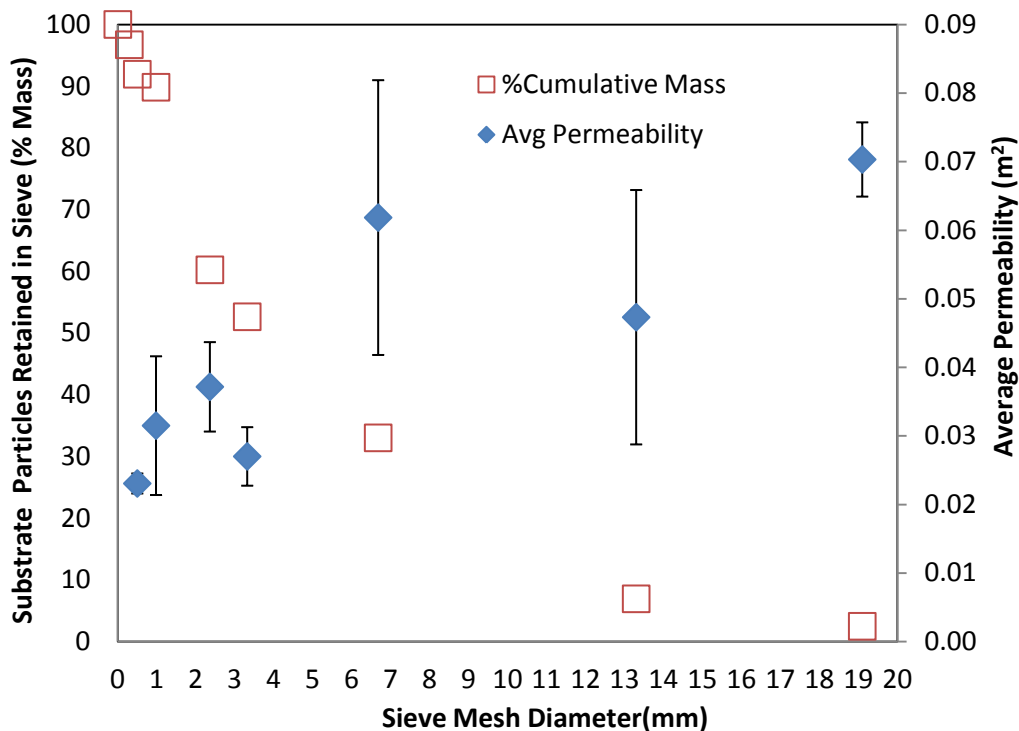


Figure 3.8. Effect of Fragment Size on Permeability where Fragments Retained in the Sieve Were Loaded Into the Column (Hanif 2012)

It is possible small manure fragments are the cause of clogging when liquid is supplied to manure columns (Svennson et al. 2007, Fleming and Cullimore 1999, and Yu and Rowe 2011). The particulates fall into the pore space, blocking liquid flow. This can take years (Fleming and Cullimore 1999), but these experiments were completed in a matter of weeks. Another possibility is that the small fragments are necessary as easily degradable material in order to grow a microbial population that can break down the large fragments during liquid flow through a column. Again, liquid flow experiments would need to be conducted to determine if this is the case.

3.4 Conclusions

Based on previous research and the permeability data, straw is recommended as a bulking agent. Wood chips do not show potential to increase the flow through a column of manure. Fragment size did impact k , however the difference was not statistically significant ($P > 0.05$) and removal of small fragments requires removal of a large percentage by mass of the manure. This approach does not show promise for large scale application. Compression was the main contributor in change to k values.

A better system should be implemented for the intrinsic permeability tests. If the apparatus is left without use for several days, many parts have to be reconstructed. The water in the column is unpredictable. At times it would shoot out the top of the system, and others it would not. A pre-compressed water column should be utilized that is continuously filled with fresh water. A monometer allows water to flow through it easier should also be implemented. The entire experiment takes almost an hour to reach equilibrium.

4.0 MEAN RESIDENCE TIME EXPERIMENTS

4.1 Introduction

The experiments conducted to determine intrinsic permeability using compressed air flow through manure columns provided some useful preliminary information about approaches to improve liquid flow through manure. However, experiments also need to be conducted with liquid flow in manure leachate columns to validate that data. Based on the data collected in the intrinsic permeability experiments, it was decided to utilize a compression of 49 J/m^2 , and that bulking agents (straw in particular) were a possible option for increasing intrinsic permeability. In this chapter, water was applied to manure columns via trickle flow and manure was not completely saturated. Data on the performance of the leachate bed reactor in terms of efficiency of organic solubilization into leachate are presented by Asma Hanif in her M.S. thesis. The focus of work presented here is to evaluate the hydraulic conditions in leachate bed reactors.

Tracer tests give an indication of the time it takes for one molecule of water to leave the system. This is called the mean residence time (\bar{t}). The experiments also give an indication of the flow regime through the column. The presence of dead zones and channelized flow can be identified based on these tests. Tracer tests were conducted at various times (Day 2, Day 9, and Day 53) of the 60 days of operation. Leachate bed reactors were packed with manure to evaluate potential changes in hydraulics over time.

4.2 Methods

4.2.1 Experiment Setup

Three columns filled with only manure (no bulking agents were utilized), a 20-mm layer of sand on top, and 40-mm layers of gravel at the bottom and top of the column (see Section 4.2.1.2 for specifics on column loading) were used in the following experiments. The experiment started with

water that had a low oxidation reduction potential (ORP) (-300 to -500 mV) that was trickled through a column of manure (see Section 4.2.1.3 for creation of low ORP water). After the low ORP water passed through the column successfully, the hydraulic characteristics were analyzed. The analysis included tracer tests performed throughout the experiment duration, liquid holding volume, and free drain volume both of which were performed after the duration of the entire experiment.

4.2.1.1 Sample Collection and Homogenization

During the course of the year of this study, Five Rivers purchased a large chipper (Bently Agrowdynamics). A chipper such as the one applied here would likely be applied in large scale applications of a leachate bed reactor. Manure in the field can vary from microscopic diameters to several meters in diameter. Samples for this experiment were collected in August of 2011. Samples were collected from a pile at Five Rivers Cattle Feeding that had been chipped, and were not necessarily representative of the entire feedlot unlike samples collected for the intrinsic permeability experiment (Chapter 3). A pile had been created of already chipped manure, but the owner could not identify where it came from. Therefore, the duration of manure storage prior to sampling is unknown. The duration of storage may have impacted ease of biodegradability, where manure stored for a longer period may have had less biodegradable constituents. Using this manure from the chipper, however, decreased sorting time from one week for 20 buckets to 5 h for 60 buckets, since there was no need to separate the small fragments and pulverize the rest. The product from the chipper was manure fragments smaller than 50-mm (2-in) in diameter. The sample was distributed into 4 equal parts by weight. Each section was evenly distributed among 5 gallon buckets. Each bucket was considered homogeneous and representative of the entire chipped pile.

4.2.1.2 Column Construction

The mean residence time experimentation apparatus columns had a similar structure with two removable caps as described for the intrinsic permeability experiments (Section 3.2.1.3). However, the

columns were made from Plexiglas. One advantage of these columns is that they are transparent, enabling visual observation of experiments. The caps were not made of PVC, but were a rubber ring enclosed by a yellow plastic cap (caps by Cherne® 8" Gripper Mechanical Plug #270-288 patent # 4,493,344). A large bolt was placed in the middle with a wing nut to tighten the cap. Five holes were present in the top cap to create an even distribution of water (see Figure 4.1 and 4.2). Each hole had a bulk head with a needle valve. The bottom cap only had two holes. As in the IPTC columns, one of the holes was closed at all times. The columns measured approximately 7.46-m (3-ft) tall with a diameter of 0.2-m (7.9-in).

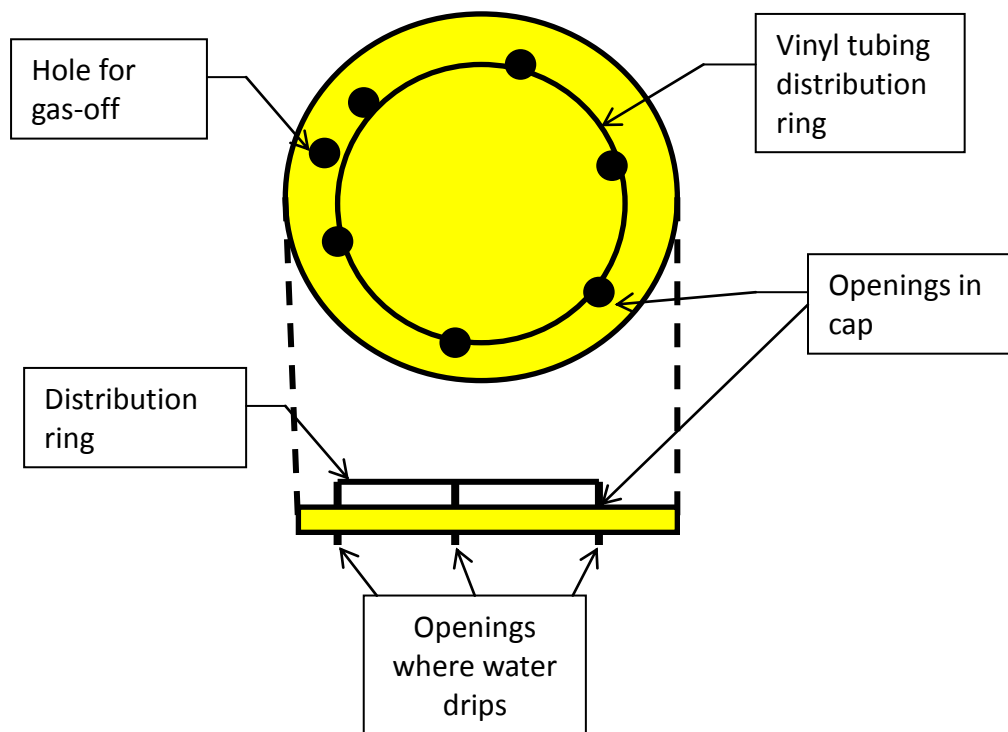


Figure 4.1. Cap Diagram



Figure 4.2. Top Cap Inside a Column

4.2.1.3 Low Oxidation/Reduction Potential (ORP) Water

Clean water added to the leachate bed reactor needed to be anaerobic to adequately represent the conditions in an actual leachate bed reactor operating as part of a multi-stage anaerobic digester for processing manure. The apparatus used in this experiment attempted to eliminate as much dissolved oxygen as possible from water, while not adding so many chemicals that the anaerobes would not survive. A combination of nitrogen gas and aluminum were used to reduce the ORP (Figure 4.3). The process started by heating reverse osmosis (RO) water (Siemen's RO Filters) to 40°C (Heater by Tempco). The RO reservoir was approximately 9.2 m (30 ft) above the heater. The RO water was siphoned to the heater. At the outlet end of the heater was a 12.7-mm (0.5-in) chlorinated polyvinyl chloride (CPVC) pipe that ran up to the same grade as the RO reservoir. Just after the heater, nitrogen gas (Organomation Associates, Inc. N-EVAP™ 111 Nitrogen Evaporator) entered the CPVC pipe for two purposes: to sparge some of the oxygen out of the water as well as lift the water back up to the RO elevation. A 12.7-mm (0.5-in) CPVC tee was inserted 6.1 m (20 ft) above the heater so the unutilized nitrogen gas could escape to the atmosphere as well as the sparged oxygen. From the teed off line, a 12.7-mm (0.5-in) barb was placed for a vinyl tubing line.

Approximately 6.1 m (20 ft) above the heater, the 12.7-mm (0.5-in) vinyl line filled with heated, nitrogen sparged water, entered a 203.2-mm (8-in) diameter Sch. 40 PVC pipe with a Sch. 40 PVC cap on one end. The cap had two 12.7-mm (0.5-in) barbs. The vinyl line connected to one of the barbs at the bottom of the cap. This barb connected to a 19-mm (0.75-in) CPVC tube. The water would come up through the tube and react with 0.1 M sodium hydroxide (NaOH) to raise the pH. The second barb at the bottom of the cap connected to another 203.2-mm (8-in) diameter PVC tube with a cap that had only one barb at the bottom. The water entered the bottom of the second cap and immediately came in contact with non-valent aluminum. When introduced to warm and high pH water, the aluminum created aluminum hydroxide (AlOH), taking the remaining dissolved oxygen out of the water. The final

step before the outlet was to neutralize the pH with 0.1 M hydrochloric acid (HCl), dosed in at the same rate as the NaOH. The water maintained an ORP of approximately -300 to -500 mV and a pH between 7.5 and 8.0. The idea and much of the construction of the ORP tank was derived from Lucas Loetscher.

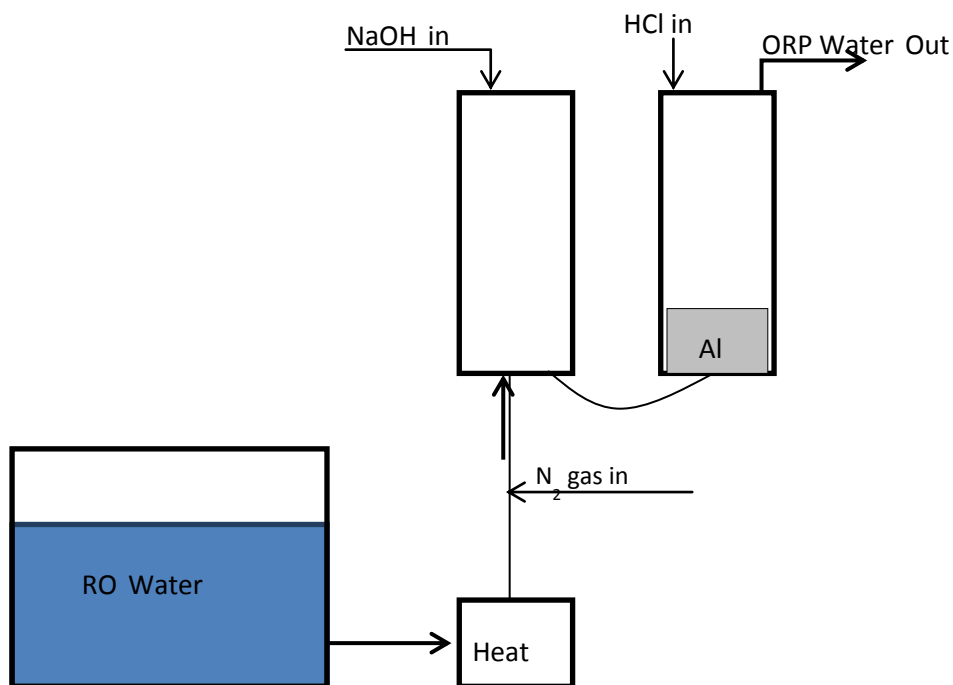


Figure 4.3. Water ORP Reduction

4.2.1.4 Plumbing

From the second column in the ORP reactor, the water was siphoned back down to the heater. From there, it had one of three fates: a primer line, a nutrient-dosed line, and a direct line to the columns (non-nutrient dosed). Some columns were dosed with nutrient and some not (for more information, see Asma Hanif's thesis), however the focus of data reported here was on the columns without nutrient dosing. To create the three influent tubes, the original tube had a tee to create the priming line. The priming line was used to recreate the siphon if the water was turned off for some reason. A second tee was then placed to distinguish the nutrient-dosed from the non-nutrient dosed line (Figure 4.4). All plumbing lines were Kuritech 6.4-mm (0.25-in) vinyl tubing.

The common manifold influent line entered through a raised floor to the temperature-controlled room. The room was made from insulation with an R rating of 6.5 (Therma Sheath-3), measuring 2.4 m^3 (8 ft^3). Figure 4.5 shows the 25.4-mm (1-in) Sch. 40 PVC supports with 3-way tees in the corners used to maintain the structure of the insulation room. An extra support ran across the ceiling. The floor of the room was a piece of insulation on the bottom with a 12.7-mm (0.5-in) thick piece of plywood. The room floor was nailed to the lab floor for safety. In two places, the floor had a hole: one for all electricity and water inlets, another for water and electricity outlets. This created a temperature efficient room. Two space heaters were placed on either side of the columns and set to maintain a temperature of 95°C . The heaters had internal thermostats that kept the temperature constant. A light was also kept in the room. Both the heaters and the light were kept on at all times.

Columns were placed on a platform approximately 0.3 m (1 ft) off the ground. The platform had a support for two sets of columns with a hole between the supports for drainage. A set of three columns was set on each support: nutrient dosing and non-nutrient dosing. The main influent tubing containing the water entered the room through the raised floor and entered a distribution manifold to evenly distribute the reduced ORP water to each of the three columns (Figure 4.6). Immediately following the manifold, each influent tube was equipped with a tracer injection line in which the ORP water could be turned off, and the tracer injection could easily be injected into the columns. After the tracer lines, each line went through a rotameter to read the flow rate of water (Figure 4.5). The rotameters were read by the operator and used to adjust flow to maintain a 2 mL/s flow (hydraulic loading rate of 0.88 m/d) with a needle valve. The water then entered the even distribution caps at the top of the columns.

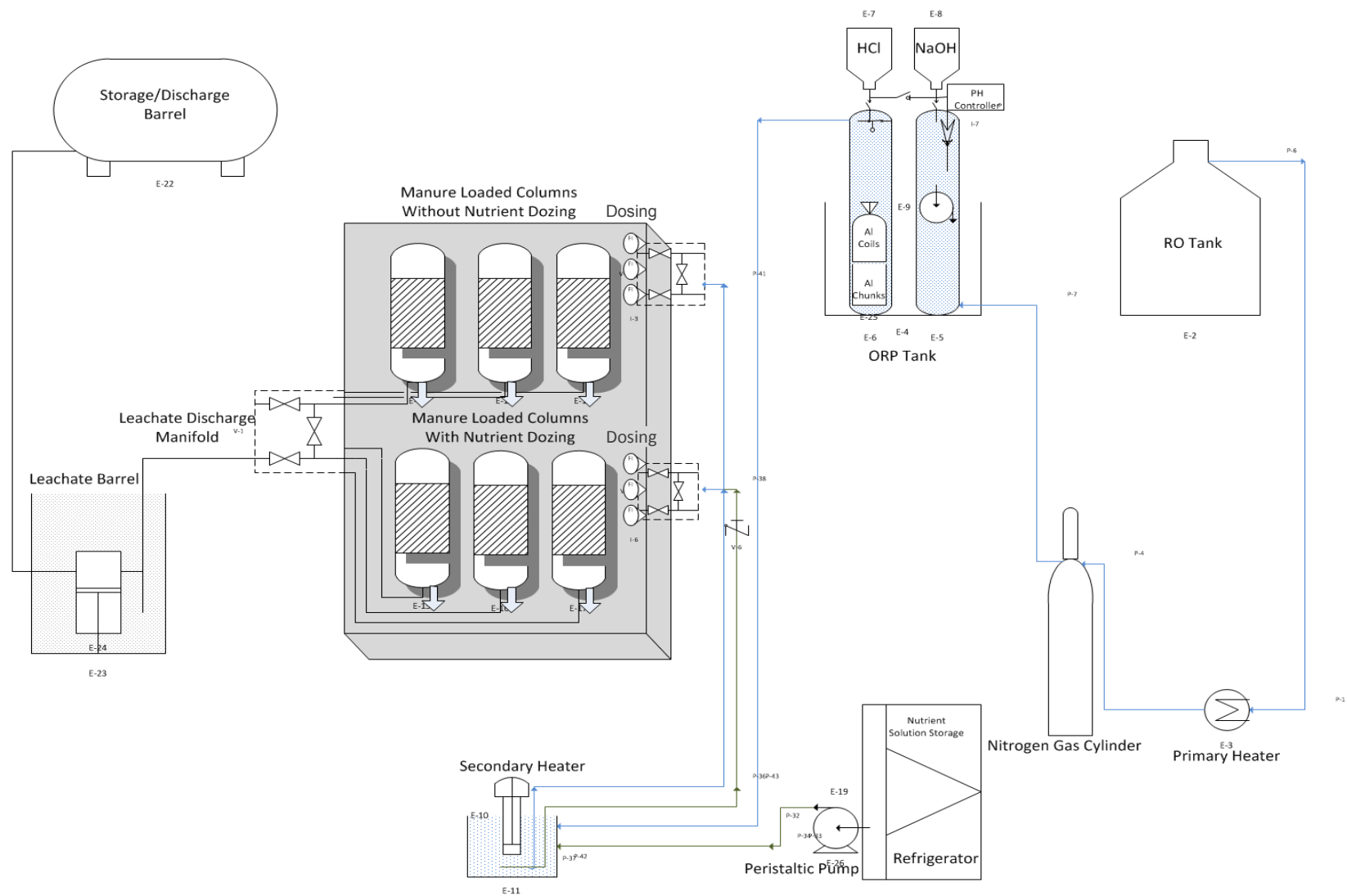


Figure 4.4. Schematic of New Technology (Hanif 2012)

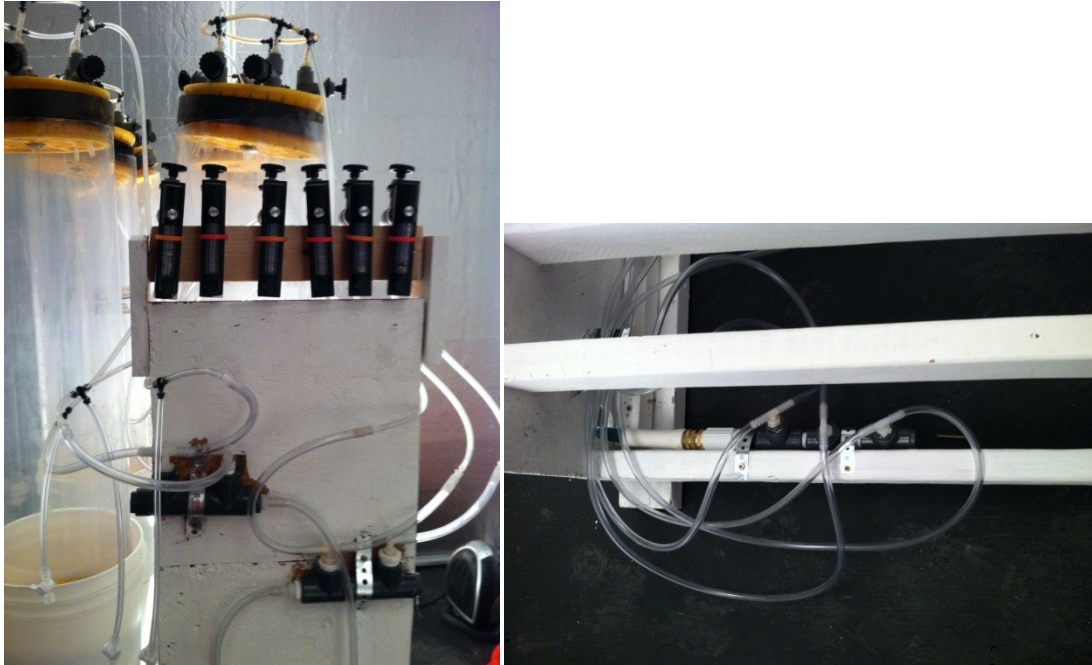


Figure 4.5. Inlet Manifolds, Rotameters, Inlet Distribution Caps (Left) and Outlet Manifold (Right) (Hanif 2012).



Figure 4.6. Outside of Temperature Controlled Room (Left) and Inside with PVC Supports (Right) (Hanif 2012).

Water trickled through the column (containing manure) and exited the bottom cap. The 6.4-mm (0.25-in) tubing from the outlet had a 3-way valve to allow the sample to be dumped, or collected in a plastic carboy. After the valve, the effluent vinyl tubing lines each entered another manifold to bring all the effluent lines to one hose. The hose exited the room through the raised floor to a drainage basin

below the temperature controlled room. Inside the basin was a sump pump, which moved water to a 1,000-L storage tank. Periodically, the storage tank would be emptied by gravity to another storage tank on a truck and emptied at Colorado State University's manure storage facility located on the Foothills Campus. The majority of construction on the temperature controlled room and plumbing was done by Asma Hanif.

4.2.1.5 Column Loading

The columns were loaded with manure (no bulking agent) as described for intrinsic permeability experiments (Section 3.2.1) with 49 J/m^2 of compression applied to each 100 mm lift. Figure 3.3 can be used to show the compression apparatus. The apparatus had similar characteristics to the IPTC columns described in Chapter 3, just on a larger scale: a wooden circle to fit the inside of the 192-mm column as well as a pole to support the apparatus, a PVC pipe with string and a weight. The number of drops as well as the J/m^2 were kept consistent between the columns discussed in Chapter 3 and Chapter 4. To maintain these parameters, a different height and weight was used in the columns discussed in Chapter 4. In Chapter 3 a weight of 1.558 kg were lifted 127 mm while 2.646 kg was lifted 170 mm in Chapter 4. All columns described in this chapter were exposed to 49 J/m^2 .

After several failed attempts at liquid flow through a column with only gravel at the top and bottom of the columns, a new approach was successfully implemented (see Figure 4.7). Three columns were used for analysis to create triplicates. Each column had an even distribution cap to allow flow through the entire column. Beneath the cap lay 40 mm of gravel. Beneath the 40-mm of gravel lay 20 mm of sand to enhance liquid distribution of flow prior to entering the manure column. The gravel was Quickrete Gravel in a 22.7 kg (50 lb) bag purchased from Home Depot with an average diameter of 10 mm. The "sand" was a crystallized glass with uniform diameter. A 50 mm² mesh (MSC Supply) was placed underneath the sand layer to allow more liquid distribution. The columns had 0.70-m of manure that was compacted with 49 J/m^2 of energy in lifts of 100 mm. Each lift had a layer of 50 mm² mesh

between it to ensure structural stability and allow for flow pathways within the substrate. Bulk density was not measured because the compressed samples were impossible to extrude from the column without changing the energy applied. At the bottom of the manure were another 40 mm of gravel, followed by a final 50 mm² mesh to prevent gravel entering the effluent tube, and a single outflow for leachate collection (Figure 4.7).

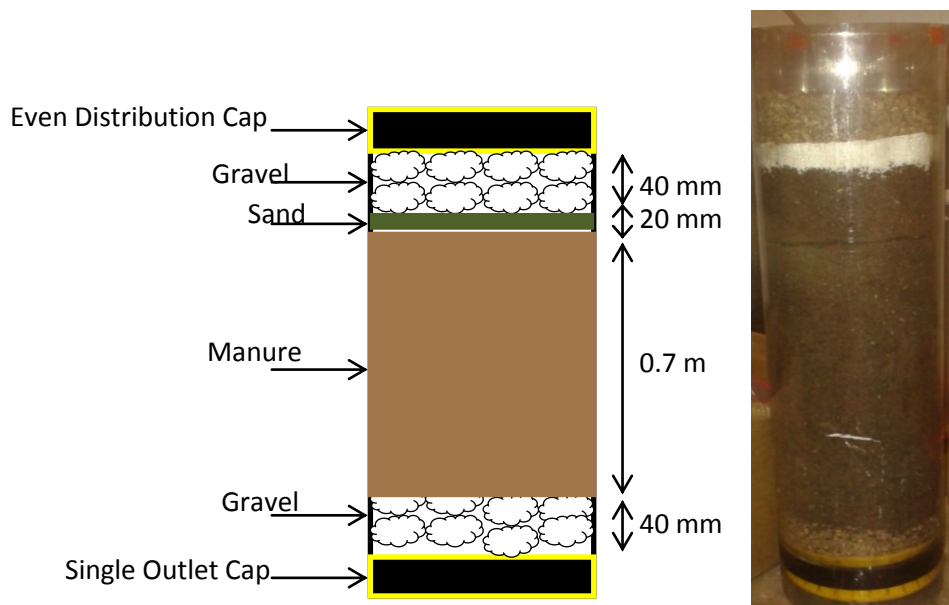


Figure 4.7. Schematic and Photo of Column

4.2.1.7 Tracer Tests

Three tracer tests were conducted through the course of the 6 week experiment. One was done the second day of the experiment to try to gain an understanding of the hydraulics shortly upon initiation of liquid flow through the columns. The second was performed a week later on Day 9. The third tracer test was done once it was determined no more digestion could occur in the columns based on stabilization of the effluent COD content (Day 53).

Rhodamine dye was attempted in a previous tracer test, but the dye absorbed too much to be effective as indicated by very low mass recovery of rhodamine input. The rhodamine dye was used in 9 tests (3 triplicates). Sodium Bromide (NaBr) was used instead. This decision was made based on

previous experiments on landfill leachate systems (Oni et al. 2009). The bromide salt was dissolved to a concentration of 5 g/L in RO water. A 10 mL dose was added to the tracer injection line for a mass addition of 50 mg. Usually potassium bromide is used in tracer tests to ensure the sodium will have no negative impacts on the hydraulics of the substrate. However, potassium bromide was not as readily available and the original concentration of sodium in manure is 1.1 g/L (Fang et al. 2011). This means the initial mass of sodium was approximately 1.02 kg (2.25 lbs) in one column of manure. Only 50 mg were added in each tracer test. Such a small plug followed by 20 mL/min of reduced ORP water for 36-h suggests the impact of the sodium was negligible. However, it is recommended that all future experiments should be conducted with potassium bromide.

The injection was monitored over a 36-h period. The duration of monitoring was based on 15 (5 triplicates) preliminary tests that showed a minimum of 60% of the mass injected or more leached. Bromide is present in the waste material, and to account for this a leachate sample was collected the night previous to the experiment for estimation of a baseline concentration of bromide. During the first 3 h, samples were collected every hour. For the following 6 h (hours 3-9), samples were collected every 30 min. This was done so that a large number of samples would be available near the expected time of peak concentration leaching. After hour 9, the frequency returned to every hour for another 3 h (hours 9-12). At hour 12, the frequency decreased to samples every 2 h for another 2 samples (hours 12-16). From hour 16 to the end of the test (hour 32) samples were collected every 4 h. For the third tracer test (Day 53) samples were collected every hour for the first 8 h, and every 2 h until hour 14. After that, sampling followed the pattern described above.

In previous experiments, water was observed to exit the columns in periodic plugs rather than at a consistent flow rate. Therefore, instantaneous samples were deemed not meaningful and samples were collected as composites instead the samples were collected over the entire time between sampling times as opposed to instantaneous sampling. The impact of inconsistent flow is that the

effluent concentrations become variable. That is to say varying effluent flow rates flush the tracer inconsistently, rendering instantaneous sampling obsolete. The observed flow out of the columns was not the same as the flow supplied to the columns. By compositing the samples, the variable concentrations were compiled over time to evaluate whether the columns do not experience drastic differences in concentration over the 36 hour period.

At each sampling interval, the volume leached over the sampling period was recorded, thus providing an estimate of flow out of the columns. The conductivity was also taken to estimate how much salt had exited the column. The bulk sample collected over the sampling interval was collected in a carboy under the 3-way valve. The carboy was then weighed, shaken to ensure a mixed sample, and a 10-20 mL sample was placed in a sample tube to be analyzed for bromide. After the tracer test, samples were stored in an 11°C room until they could be analyzed using ion chromatography (IC) (Metrohm 861 Compact IC with a 0.45µm filter).

Tracer tests were conducted in three replicate columns. All samples from the Day 2, Day 9, and Day 53 tracer tests from Column A were analyzed to gain a detailed understanding of what had occurred. All of the samples from Column A, Column B, and Column C were analyzed by IC in the Day 53 tracer test. In order to not waste resources, ten samples (45% of the 23 samples collected in the 36-h experiment) were analyzed from Column B and Column C for the Day 2 and Day 9 tracer tests. To figure out how much of each sample to add to the IC injection number, the total volume leached over the time interval was calculated. The percentage of volume of each individual sample was calculated as a percentage of the total volume leached for each time interval. This volume was the percent of each individual sample that was added to the IC injection number and run through the IC machine.

Residence time distribution (RTD) curves were calculated based on tracer test data where $E(t_{Mi})$ is the probability that 100% of mass applied will exit over the sample time interval (Equation 4.1)

$$E(t)_{Mi} = \frac{Q_i * C_i}{M_T} \quad 4.1$$

where: $E(t_{Mi})$ = residence time distribution function (h^{-1}), t_{Mi} = midpoint time in the composited sample time interval (h), Q_i = flow over the sample time interval (averaged because it is a composite sample) (L/h), C_i = concentration of sample collected over sample time interval (mg/L), M_T = Total mass exiting the column during the tracer (mg).

These equations differ from traditional RTD determination in that typically the concentration at a discrete time t is utilized. Here, samples were composited due to high variability of effluent flow rate. Based on $E(t)$, \bar{t} can be calculated by Equation 4.2.

$$\bar{t} = \sum E(t_{Mi}) * t_{Mi} * (t_{Mi} - t_{Mi-1}) \quad 4.2$$

where: \bar{t} = mean residence time, $E(t)$ = residence time probability distribution, t_{Mi-1} = time at the midpoint of the previous sampling interval.

Figure 4.8 demonstrates the observed surges in effluent flows. The point where the sample is shown on the graph is the midpoint in the time interval of the composite sample. A plastic carboy (approximately 22.7 L (5 gallons)) was utilized to collect the effluent from the columns. The 3-way valve at the effluent was turned from draining to the effluent manifold to draining for sampling. At the end of each time interval, the carboy was weighed. It was assumed that the leachate had the same density as water. This is how the volume leached was calculated.

After each carboy was weighed, a sample was taken for IC analysis. The carboys were then emptied into the drainage basin where regular flow ends. Each carboy was rinsed with tap water before being put back under the column. While the carboy was being sampled, emptied, and rinsed, another carboy was placed under each column so in order to avoid losing any flow or tracer.

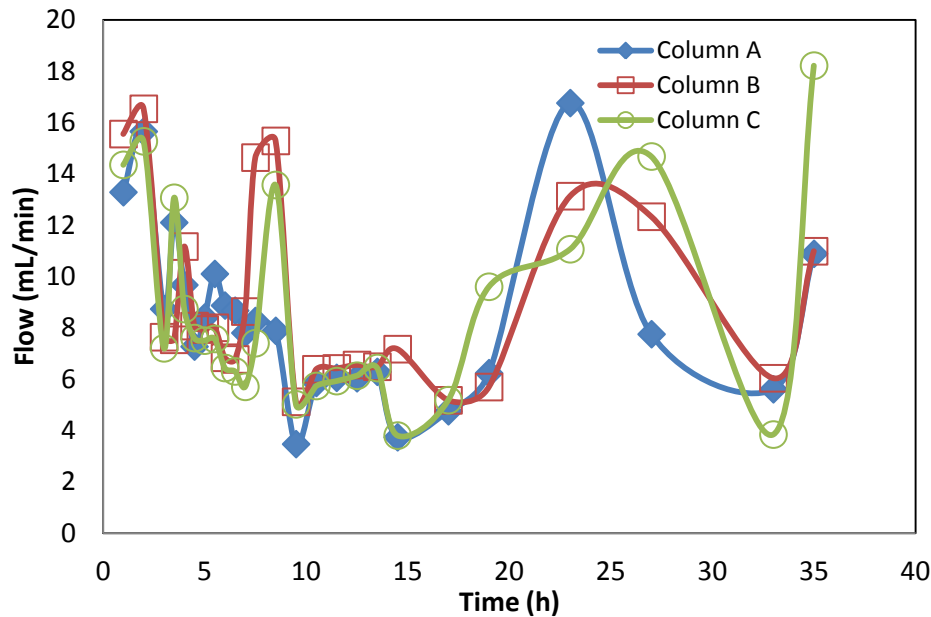


Figure 4.8. Day 2 Effluent Flow Over the 36 Hour Experiment

The data were also analyzed based on the percentage of the tracer plug injected exited the column. It was decided that the cumulative mass would be evaluated. The way this was calculated was the total mass that was injected, called Mass I, was assumed to be 100% of what could possibly exit the column. The mass of the first time interval (concentration reading from the IC multiplied by the effluent volume during that time interval), called Mass X, was added to the mass of the second time interval, called Mass Y. The sum of these numbers was called Mass Z. Mass Z was then divided by the Mass I to give the percent accumulated in the effluent over the entire time period. The mass of the third interval, Mass N, was then added to Mass Z to create Mass F. Mass F was then divided by Mass I to get the percentage of total injected mass that had exited the column up until the end of the third time period. The process continued until the all of the time intervals were added. The final percentage is how much mass leached from the column divided by how much mass was initially injected.

The hydraulic loading rate (HLR) remained constant throughout the entire experiment. Equation 4.3 shows how HLR is a function of flow into the column and the cross sectional area to receive that flow. The HLR is representative of the velocity of water that moves through the column.

$$HLR = \frac{Q_{in}}{A} \quad 4.3$$

where: HLR = (m/d), Q_{in} = flow of water into the column (20 mL/min), A = cross sectional area of the column (0.032 m^2). Equation 4.4 shows the calculation of the ratio R . This ratio is a ratio of the superficial velocity to the HLR. The HLR (0.88 m/d) is representative of the velocity of water to the column assuming the flow is distributed through the entire cross sectional area of the column. If the ratio R is greater than one, the flow distribution is evenly distributed. If the ratio R is less than one, the flow is not evenly distributed and moves rapidly through the column due to preferential flow paths.

$$R = \frac{SV}{HLR} \quad 4.4$$

where R = ratio R and SV is the superficial velocity. The SV is calculated in Equation 4.5. It is a function of the mean residence time for each tracer test and the length in the column. This calculation assumes uniform flow through the column.

$$SV = \frac{\bar{t}}{L} \quad 4.4$$

Where L = length of the column (m).

4.2.1.8 Free Drain Volume

An experiment was designed to understand how much volume will drain after water is no longer being delivered to the system. This helps to understand how much liquid flows through the system during operation. Water was turned off at the end of the six week period. The water leached out was collected until no water drained in a 30 minute period. This experiment ran for 55 h and 12 min. That is to say it took 55 h and 12 min from the time water stopped being delivered to the column to when it stopped draining for a 30-min period.

4.2.1.9 Liquid Holding Volume

Liquid holding volume was determined to understand how long it takes ponded water to drain through the column. The 3-way valve was shut off and the columns were filled with water. The water ponded approximately 40 mm deep and was allowed to drain so that each column had water flowing

out the bottom, or the entire column was wet and draining. The 3-way valve was closed a second time and the columns were once again filled with water to approximately the 100 mm mark above the gravel. The valves were turned to flow to a carboy, and the water was allowed to exit the column. The height difference was calculated over time (see Figure 4.9) by drawing a line directly onto the column and documenting the time the line was drawn. The volume leached out over the experiment was recorded.

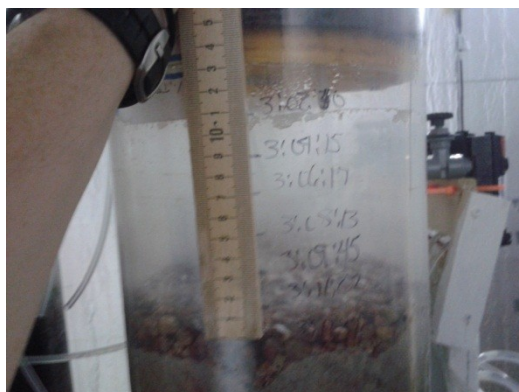


Figure 4.9. Free Drain Volume Experiment

4.3 Results and Discussion

4.3.1 Tracer Tests

Liquid distribution media (sand and gravel combination) proved to dramatically enhance flow. Four full scale experiments were put together previous to this experiment set and failed within a week prior to the addition of sand. Other researchers have struggled to maintain flow through manure for more than a week (Myint and Nirmalakhandan, 2009). Here flow was sustained for six weeks. The average influent flow was maintained at a constant 20 mL/min. The sand created a dispersion surface that was essential for the manure to accept the water. Further investigations are necessary to fully understand how this worked. Visually the sand dropped into channels at the commencement of water flow. It appeared as though this was the beginning of channelized flow. The manure seems to need a highly permeable substance to get water flowing due to variability along the wetting front (Coppola et al. 2009).

Channelized, or non-uniform, flow occurred in every column and every test as shown in Figures 4.8 and 4.10. Figure 4.8 shows the initial tracer test, and the Figure 4.10 shows flows for the next tracer a week later. The effluent flow is much larger during the Day 9 tracer test compared to the Day 2 tracer test. This could be attributed to start up conditions. The column was wet, but not every flow path had been utilized by the time the tracer started. This could also be preferential flow; the water may have been trapped in various pores and needed more time to flow through.

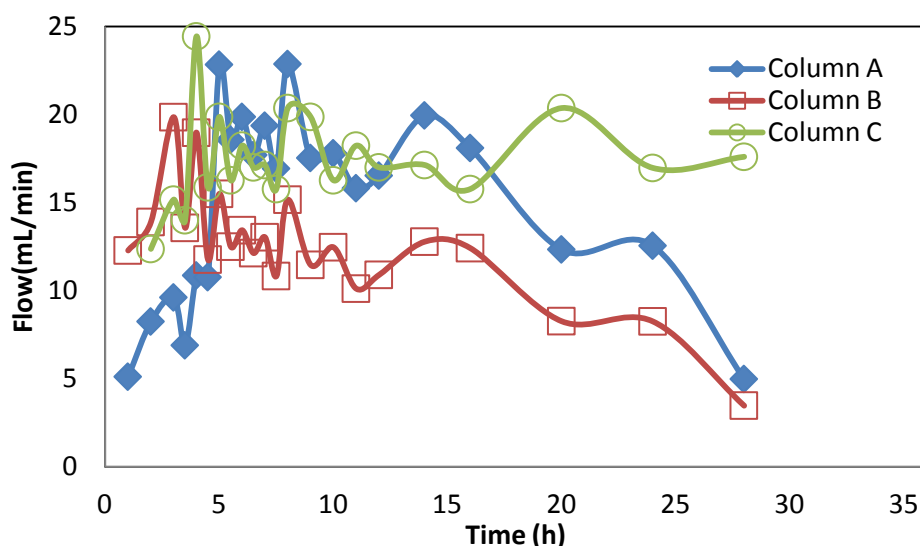


Figure 4.10. Sporadic Flow Through Each Column During Day 9 Tracer Test

Tailing of the curves was noted in all cases (Figures 4.11A, 4.11B, 4.12A, and 4.12B). Such tailing is characteristic of plug flow with dispersion. Tailing here is also likely a result of channelized flow and interaction between free flowing liquid and dead or stagnant zones, indicating preferential flow channels (Coppola et al. 2009) over the course of the tracer study.

According to Coppola et al. (2009), preferential flow has four characteristics: (i) preferential flow in real macro-pores, (ii) preferential flow in inter-aggregate pores, (iii) fingering due to the instability on the wetting front; and (iv) preferential flow due to spatial irregularities or temporal dynamics in soil wettability. All four of these characteristics were likely in these systems. The non-uniformity of the wetting front creates a preferential pathway for the water to flow. This pathway is utilized until it

erodes and collapses. Then the column fills with water until a new channel is created. Water still drips from the previous channel, but only due to the sponge-like characteristics, or inter-aggregate pores, that still drip.

The data from the Day 2 tracer had a much higher concentration of bromide compared to the Day 9 tracer (Figure 4.11A) and inability to observe a comprehensible RTD peak in column B. Slight traces of bromide can be found in manure and flush out with the initial flush of tannins. It appears that this tracer test was conducted before the system had time to totally flush. The background bromide concentration was 14.4 mg/L in the initial leachate sample. This concentration was too high to discern elution of bromide input from the tracer test. It would appear as though the peaks are when channelized flow flushes. When comparing the concentration data to the flow data, there is a parallel between when concentration has a high peak and when flow experiences a peak. The data from this test are considered invalid. However Figure 4.10A shows the initial concentration at 0 mg/L as well as a bell curve trend.

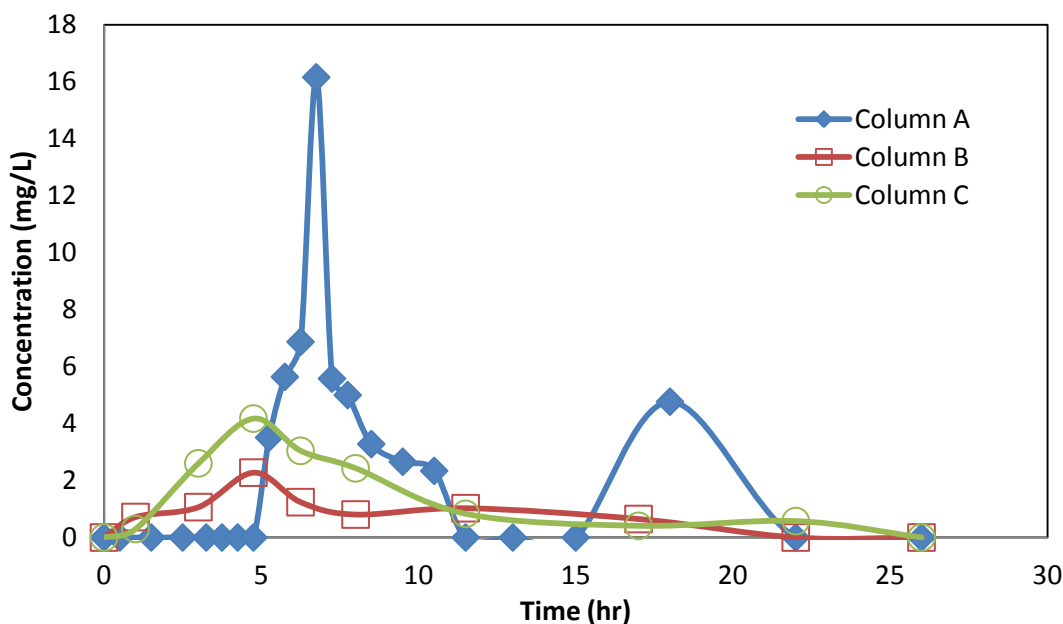


Figure 4.11A. Concentration Over Time Leached During Day 9 Tracer Test

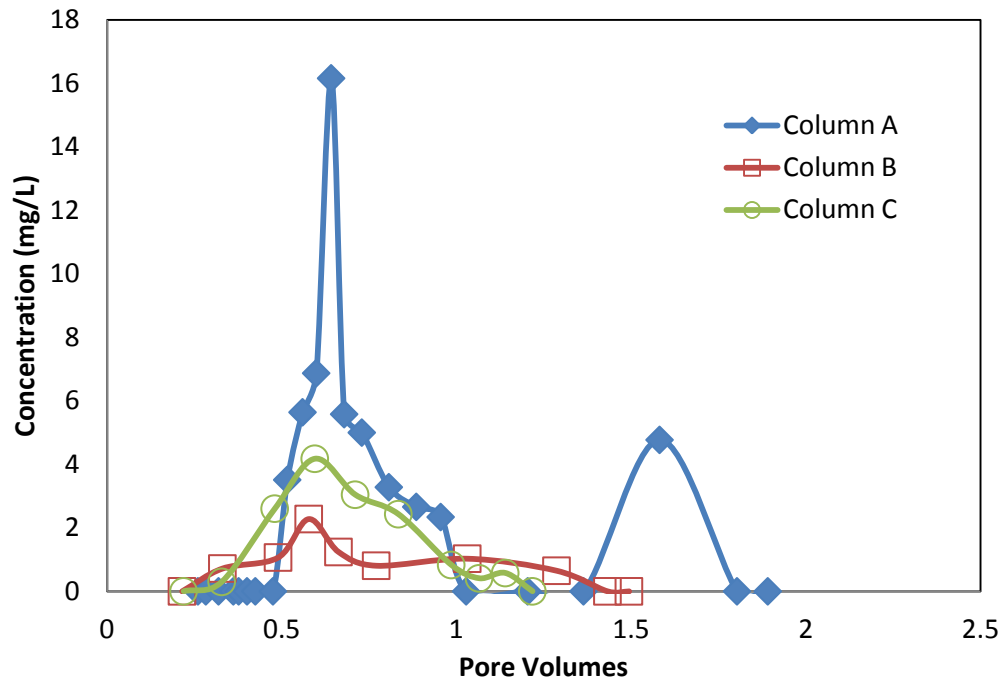


Figure 4.11B. Concentration Over Pore Volumes Leached During Day 9 Tracer Test

When observing the differences between the Day 9 (Figures 4.10A and 4.10B) and Day 53 tests (Figures 4.11A and 4.11B) one can note that a higher concentration left earlier in the experiment in the Day 53 tracer test, but the peak was not as drastic as in the Day 9 tests. The Day 53 tracer test showed that it takes many more pore volumes for the tracer to exit the column. However, when compared to the cumulative mass figures, more mass exited over the 36 h experiment in the Day 53 tests. These concepts most likely show a retardation of flow due to dead pore spaces. These areas allow flow in, but not out. In a complicated matrix such as manure, these zones are common (Rosqvist and Destouni 2000). The dead zones trap the tracer. The more frequent the dead pore spaces, the less mass of the tracer will exit the column.

When looking at the concentration plots in terms of pore volumes, it appears as though the columns experience preferential flow. The peak concentration occurs well before the first pore volume leaches from the column. However, the number of pore volumes leached to reach the maximum cumulative mass leached is more than one for every experiment. Table 4.1A shows how the peak concentration occurs before one pore volume exchange and 4.1B demonstrates how the maximum

cumulative mass leached occurs after one pore volume. This lends to the explanation that dead zones in the manure are causing a portion of the tracer to exit the column well after the peak has exited.

On that note, in the all of the tracer data there are large fluctuations in concentration after the peak concentration exits, supporting the idea that some tracer does not exit with the peak concentration. Some of the tracer appears to follow a different path that experiences a slower flow rate.

Table 4.1A. Number of Pore Volumes to Reach Peak Concentration

Tracer	Column	Peak Concentration (mg/L)	# Pore Volume Exchanges
Day 9	A	16.16	0.641
	B	3.65	1.03
	C	6.51	0.59
Day 53	A	9.32	0.23
	B	5.74	0.24
	C	2.49	0.47

Table 4.1B. Number of Pore Volumes to Reach Maximum Cumulative Mass

Tracer	Column	Max. Cumulative Mass (%)	# Pore Volume Exchanges
Day 9	A	95%	1.58
	B	29%	1.29
	C	48%	1.14
Day 53	A	89%	1.56
	B	76%	1.59
	C	34%	1.02

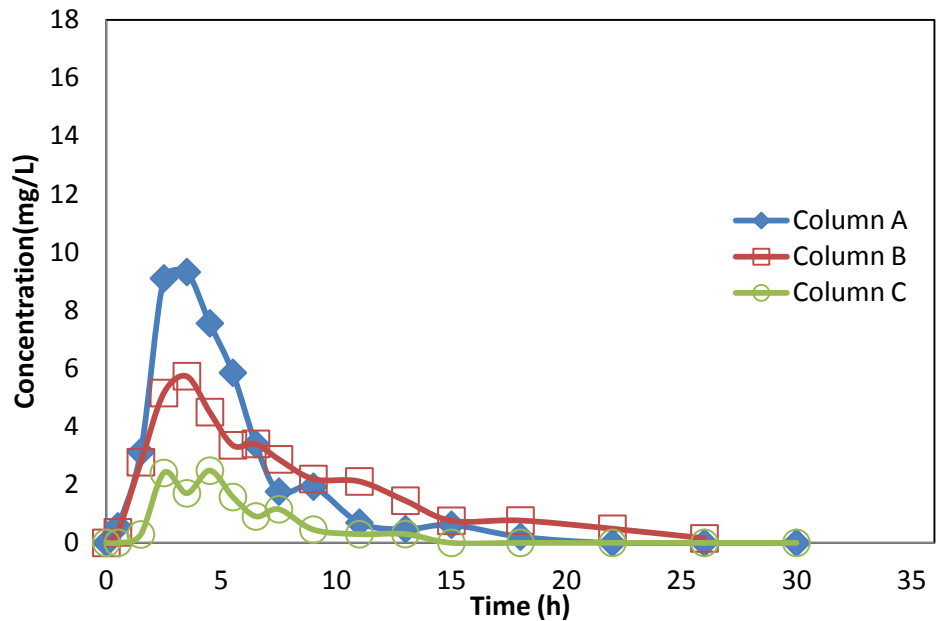


Figure 4.12A. Concentration Over Time Leached During Day 53 Tracer Test

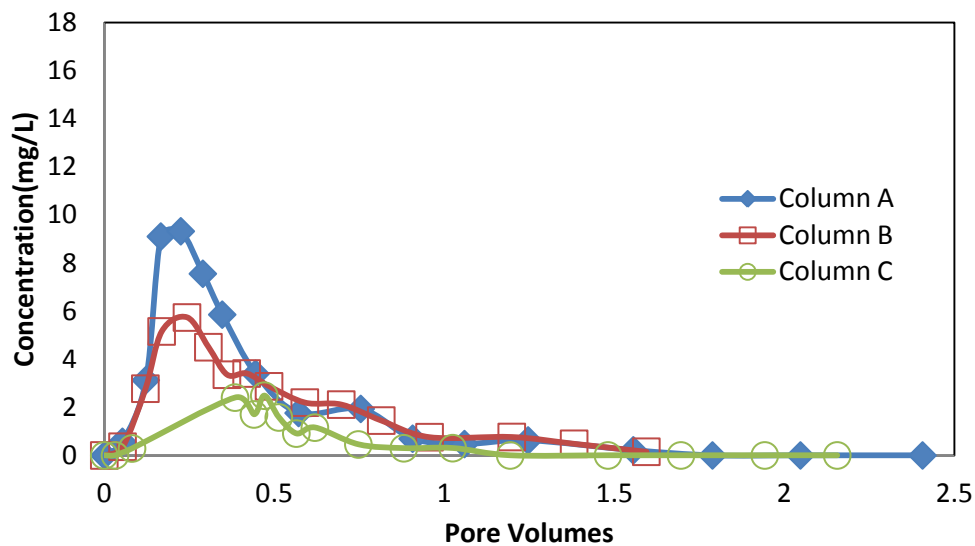


Figure 4.12B. Concentration Over Pore Volumes Leached During Day 53 Tracer Test

Due to irregular flow, the best way to demonstrate how the columns leach, is to show the percent cumulative mass that leaches over time (Figure 4.13A, 4.13B, 4.14 A, and 4.14B). Complete recovery of bromide was not achieved. The percent leached was calculated by taking the concentration over the time interval and multiplying it by the volume leached in the time interval, yielding the mass leached over a time interval. The mass was then divided by the total mass injected to the columns. The first time interval mass was added to the second interval mass and divided by the initial injection mass

and so on for all the time intervals. In tracer studies, it is rare to observe 100% of the injected mass. The percentages shown here are consistent with the literature where 55-70% recovery was achieved (Rosqvist and Destouni 2000 and Oni 2009), excluding Column B in the second tracer and Column C in the third tracer. It is expected that tracers at least 55% of tracers applied will be leached. However, due to preferential flow, it is possible that salt became trapped in a dead zone or was absorbed by an aggregate and kept in the inter-aggregate pores. These salts may take days to exit, or could stay locked up in the dead zones (Coppola et al. 2009). Given the difficulty in maintaining consistent flow through manure columns (Myint and Nirmalakhandan, 2009 and Demirer and Chen, 2008) and the inconsistent outflow observed (Figures 4.11-4.12) in these studies, the mass recovery is considered acceptable to determine \bar{t} in the columns.

Column A consistently discharged more mass over the entire 36 h experiment than any other column (Figure 4.11-4.12). This phenomenon does not start, however, until the experiment has run for 7 hours. It was the closest to the distribution manifold, which probably means it received the most consistent flow of ORP water. Columns B and C both operated with notable variability. However, they had fewer samples analyzed. Column C was the farthest from the manifold, but seemed to have received relatively consistent flows. A device ensuring continuous constant flow to the columns should be used in future studies.

The plots of pore volumes show that the majority of the tracer exited the column before an entire pore volume had exited the column. The Day 9 and Day 53 tracer tests both had their peaks occur between 0.5 and 1 pore volumes. This could possibly be attributed to channelized flow and leads to the speculation that preferential flow is present in the columns.

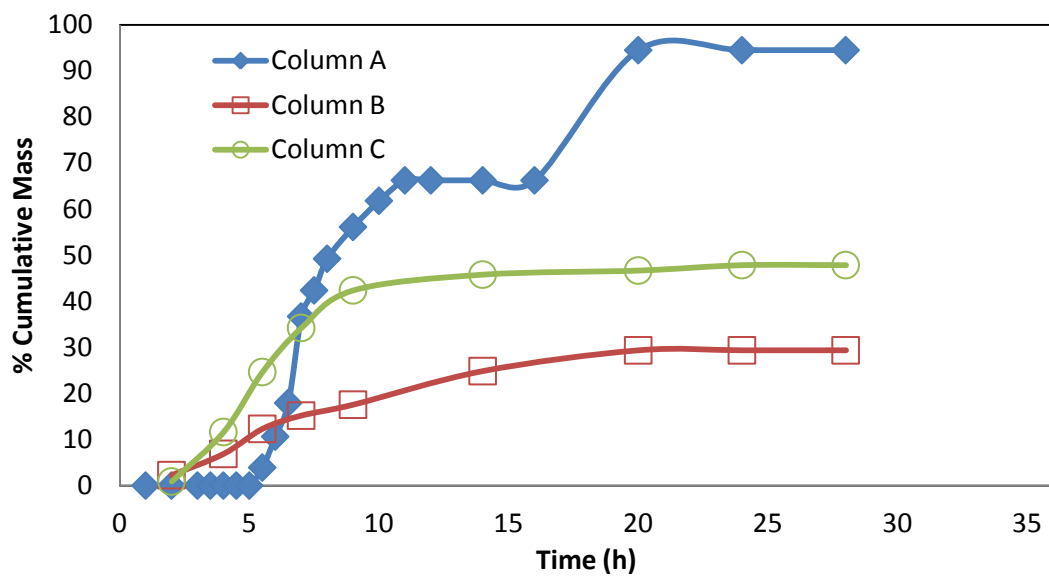


Figure 4.13A. Percent Cumulative Mass in Day 9 Tracer Over Time

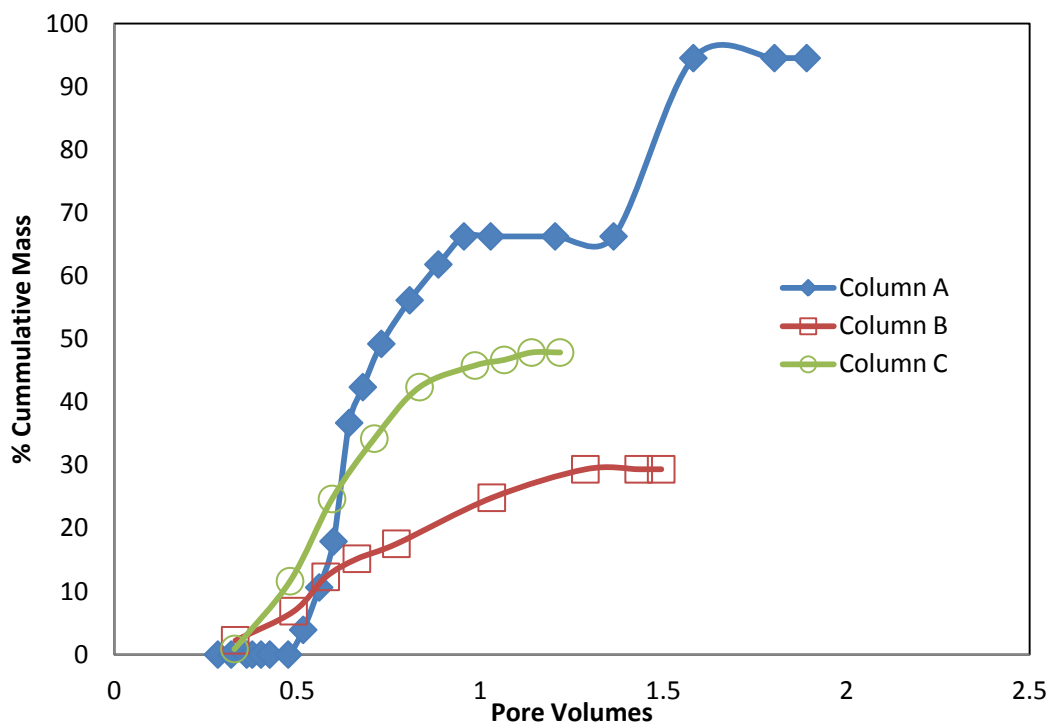


Figure 4.13B. Percent Cumulative Mass in Day 9 Tracer Over Pore Volumes

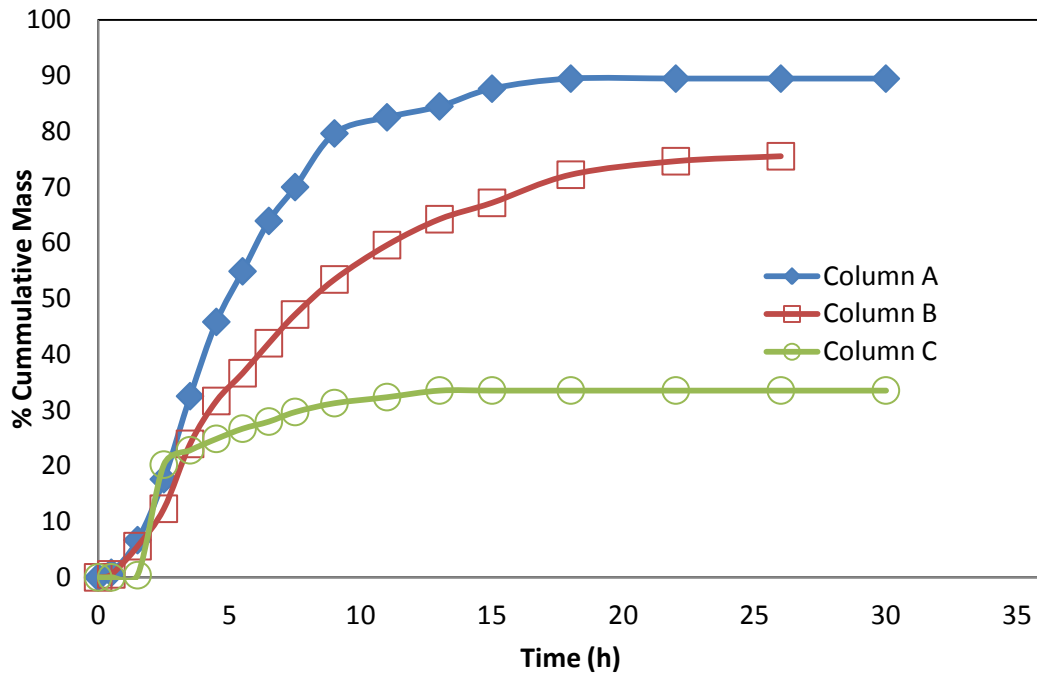


Figure 4.14A. Percent Cumulative Mass in Day 53 Tracer Over Time

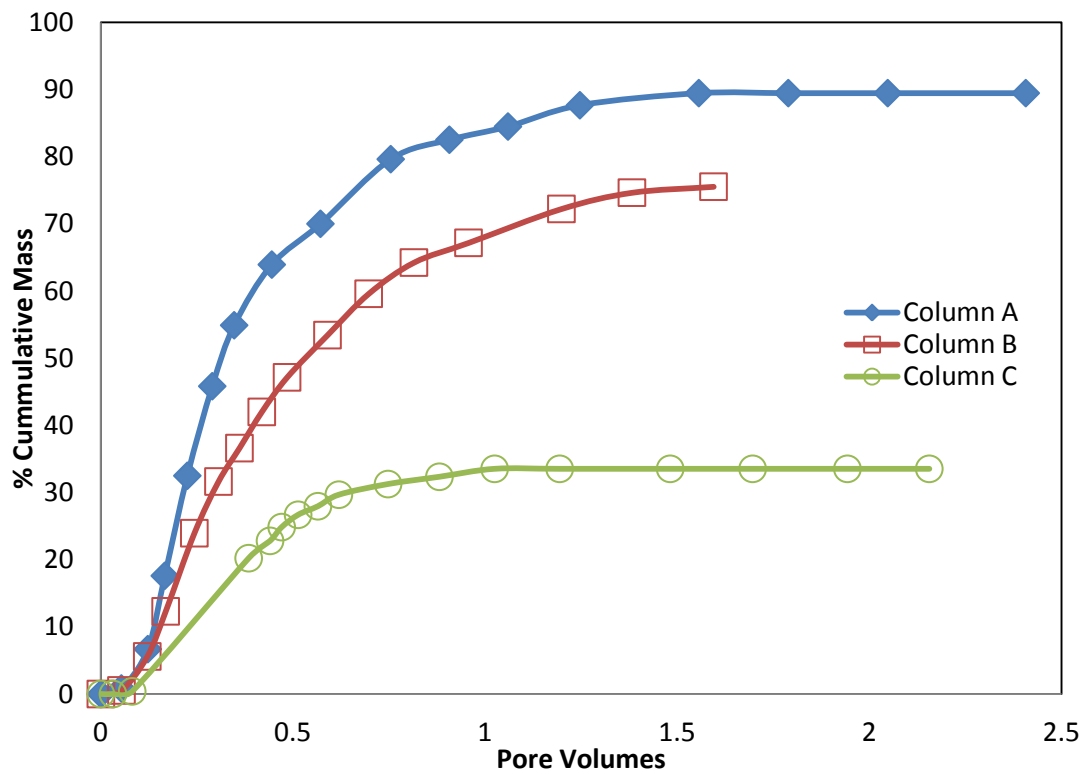


Figure 4.14B. Percent Cumulative Mass in Day 53 Tracer Over Pore Volumes

The purpose of tracer tests is to estimate \bar{t} , or the average time for a molecule of water to move through a column. Table 4.2 reports \bar{t} , calculated as explained in Section 4.2.1.7, for each column from

the Day 9 and Day 53 tracers. As mentioned in Section 4.2.1.7, if the ratio R is greater than one, the flow distribution is evenly distributed. If the ratio R is less than one, the flow is not evenly distributed and water moves rapidly through the column. Table 4.2 shows that the ratio R is greater than one for all cases, indicating that the flow is evenly distributed across the column and in many cases retarded, likely due to presence of dead zones as previously discussed. However, a small HLR was applied to the columns. It is possible with a higher HLR the columns would show a less uniform flow. Upon visual inspection flushes of leachate would leave the column in a short time frame. It is possible that the flow is evenly distributed over a long period of time, but in the interim, the column experiences a retardation in flow due to the presence of dead zones.

Day 53 had a \bar{t} value of 9.1 hours, while Day 9 had a \bar{t} value of 5.7 hours. There was also slightly more variability in the Day 9 tracer. The standard deviation is 1.8 for Day 9, while for Day 53 the standard deviation is 1.7. This shows that the time it takes for a molecule of water to pass through the column gets smaller as the experiment continues. It was thought that with biological growth and the consumption of the large fragments, the \bar{t} value would decrease over time. That is to say more biological growth would occur and clog the system over time.

Table 4.2. \bar{t} and R for Each Tracer

Tracer #	Column	Mean Residence Time (hr)	Superficial Velocity (cm/hr)	Average (hr)	Standard Deviation	Ratio R
2	A	9.0	7.5	9.1	1.8	2.035913
	B	7.2	9.5			2.555643
	C	10.9	6.6			1.77387
3	A	5.6	12.2	5.7	1.7	3.296891
	B	7.4	9.2			2.493385
	C	4.1	17.5			4.71935

A paired, 2 tail, t-test was conducted for the data from the Day 9 tracer as compared to the Day 53 tracer. The null hypothesis assumed that the Day 9 tracer \bar{t} value was significantly smaller than the Day 53 tracer \bar{t} . A t-test was chosen to evaluate the significance of the difference between the Day 9

and Day 53 \bar{t} values because the data showed a normal distribution and a null hypothesis was defined. The t-test gave a probability of 0.24, indicating that the Day 9 tracer a slightly larger \bar{t} was observed, but the difference was not statistically different at the 95% confidence interval. The difference could possibly come from channelized flow. Channels are built and over time they get larger, allowing more flow to pass through the column faster. This may not always be true, but seems to be the case in the columns tested.

Tracer injections were also difficult. The plumbing was added after the system was constructed and was not built as well as it could have been. It is possible that not 100% of the tracer entered the column. Pressure would build up in the tracer injection line, and a little bit of the tracer would get rejected and sprayed all over the heated room at the initial injection. This would partially explain low recovery values. Manure also absorbs salts well. Additionally, it is possible that some of the salt stayed in the column locked up in the aggregate or in dead zones. Unfortunately the bromide concentration was not recorded in the manure at the end of the experiment due to budgetary constraints. However, the recovery of bromide was consistent with what the literature states (Oni 2009).

4.3.2 Free Drain Volume

In trickle flow reactors it is common to determine the free drain volume. The free drain volume experiment test how a volume of water leaches through the column without water addition at the top. In Table 4.3 the data were collected over 55 h and 12 min. The average volume leached was 4.8% of the total volume in the column. The volume leached was 7.2% of the total voids in the column as determined at initiation of column experiments. The available void space at the time was unknown however. It is possible the void space decreased due to biological breakdown and shearing of larger fragments. However, these values do show how little water passed through the column with no additional water on top.

Table 4.3. Free Drain Volume Results

Column	Free Drain Volume (L)
A	0.946
B	1.036
C	0.949

4.3.3 Liquid Holding Volume

Another common test is to observe leaching in a reactor when water is placed on top, causing a large differential in the hydraulic head across the column. The columns are allowed to pond and then drain. These experiments took minutes, as opposed to the free drain volume test which took days. Data similar to those presented in Table 4.4 could be collected for columns operated in future experiments where bulking agents (straw or wood chips) are added to manure to compare hydraulics. Each column started with 20 liters of water ponded on top. Approximately 20% of the water in each column exited during the experiment (Table 4.4). The data, coupled with the free drain volume data, shows that the difference in head across the column impacts the flow of water. Water flowed faster when the column had ponded water on top. This could potentially prove important in large scale systems. Further investigations should be made into the impacts of head differential across the column.

Table 4.4. Liquid Holding Volume Results

Column	Average Draining Rate (cm/s)	Time (min)	Volume Leached (L)
A	0.021	9.42	3.942
B	0.011	22.22	3.642
C	0.014	14.02	3.852

A sealed double ring infiltrometer (SDRI) test could not be performed to measure the exact permeability because the samples could not be fully saturated. If the samples could be fully saturated with air and water, a connection between the permeability values reported in Chapter 3 could be made.

Another way to discover the missing connection between intrinsic permeability and hydraulic performance is to perform an instantaneous profile experiment. In this experiment, tensiometers are placed at various elevations on the column (Chiu 2000). These researchers tested the suction front to

estimate the permeability. However, in the experiment mentioned, the soil was uniform and poorly graded. The mentioning of uniformly and poorly graded refers to a soil that does not vary drastically in size. Manure tested in these experiments was non-uniform and well graded. The uniformity in the soil makes the wetting front uniform across the sample. In the well graded material used for these experiments, it is possible the wetting front would need tensiometers at numerous points to model the porous matrix.

4.4 Conclusion

As far as can be determined, this is the first operation of a leachate bed reactor in this laboratory containing manure where liquid flow could be sustained for more than one week. Here, flow was maintained for six weeks. The sand and gravel mixture at the top of the reactors seemed to disperse liquid flow adequately at the top of the reactor, enabling sustained liquid flow. Several hydraulic parameters were determined. These experiments provide information on the hydraulics of leachate bay reactors containing manure only. This data can be compared to data collected from columns containing manure and bulking agents such as straw or wood chips in the future.

When tracer tests were conducted most of the injected salt passed through the column in less than one pore volume exchange. However, tailing was observed in residence time distribution curves and complete mass recovery was not observed. This indicates the presence of dead zones within the manure packed columns. The ratio R was greater than one indicating uniform flow, which was unexpected. The number of pore volume exchanges was less than one for each experiment over a 36 hour period. This indicates retardation of flow, consistent with presence of dead zones. Also, only 20% of the flow exited in the equilibrium holding volume experiment. Reactor performance could be enhanced by finding ways to eliminate reactor dead zones thus improving hydraulics. Future research could address this issue. The estimated value of \bar{t} decreased over the duration of the experiment

indicating that water flows through the manure faster over the duration of leachate bed reactor operation.

The free drain volume was a small portion of the total volume (4.8%) and pore volume (7.2%). While the experiment was being conducted, it appeared as though the water was lacking a change in gradient to exit the column. It seemed as though the manure soaked up what water was in the column, much like it did when the manure created a slurry. The free drain and liquid holding volume experiments show how little water passes through the column when no water is added to the top. This lends toward the philosophy that the head differential across the column may be an important aspect to study.

5.0 SUMMARY AND CONCLUSIONS

5.1 Intrinsic Permeability Summary

In the Intrinsic Permeability Study, air was used as a media to flow through manure because manure becomes relatively impermeable when it is completely saturated. The intrinsic permeability was found using an equation derived from Darcy's Law. Manure was compressed to simulate a full scale operation where manure would be stacked in tall columns in the proposed multi-stage high solids digestion reactor. Intrinsic permeability was determined at various compressions. When manure was compressed at less than 49 J/m² permeability was highly variable. The variability in the compression and k data can be attributed to air voids. The uncompressed manure contained 31.95% air. This is also the reason behind why compacted samples have less variability. No water exited the columns during compaction, indicating the volume decrease was entirely due to the reduction of air. The main message from the compression data are that a little compression makes a large impact and a lot of compression is not necessary. Once manure is compacted, it is difficult to compact it more.

While the bulking agents increased k, the impact was not notable. Straw increased k more than wood chips. However, further experimentation is needed to understand the impact on \bar{t} . Unfortunately, there was not time to operate leachate bed reactors with liquid flow containing manure and straw combinations. Straw should be used as the bulking agent of choice in future experiments based on the k values.

While removing small fragments (≤ 4 mm) showed benefit for increased intrinsic permeability, a large portion of fragments needed to be removed (50% at a minimum) before a real benefit to permeability was observed. Hydraulic studies on manure with the smallest fragments removed could be performed to truly understand the impact on the anaerobic digestion system. However, this process is not likely to show economic benefit since most of the material would not be anaerobically digested to produce methane. In addition, operation of the leachate bed reactors (Chapter 4) showed that liquid

flow could be sustained in columns with the addition of sand at the top of the reactors. The idea of removing small fragments from manure prior to loading manure into leachate bed reactors will not be investigated further.

5.2 Hydraulic Characterization

The dispersion media proved to be the most beneficial addition to sustain long term flow. Long term flow is necessary to enable sufficient hydrolysis of manure to liquid leachate such that enough methane can be generated from this process for economic benefit. Adequate dispersion of fluid across the top of the manure column prevents slurry formation. Further investigations are necessary to fully understand how this works. The sand was observed to drop into channels at the commencement of water flow. It appears as though this was the beginning of channelized flow. The manure seems to need evenly distributed flow to prevent slurries from forming that prevent the passage of water over time. The sand that flowed into the manure column may have also helped to maintain the structure of the manure, enabling liquid flow through the system.

As far as can be determined, this is the first time hydraulic parameters for trickle flow through manure could be characterized because this is the first time flow could be sustained for more than one week. Previous experiments failed in less than one week. The free drain volume was a small fraction of the pore volume in the total column. The fact that so few pore volumes exited the column indicates severe retardation within the column that can be attributed to dead zones. Also, the residence time distribution plots show the tracer leached from the columns in less than one pore volume. This is another indication that preferential flow was present.

The \bar{t} values decreased over time. This was the opposite of what was speculated. It was thought that biological growth and degradation of the substrate would lead to less pore space and larger \bar{t} . However, the increase in \bar{t} could possibly come from channels in the manure getting larger over time and allowing more flow to pass. Tailing was observed in residence time distribution curves and

complete mass recovery was not observed. This indicated the presence of dead zones within the manure packed columns. The ratio R shows that the flow through the column was retarded because the ratio R was greater than one for every tracer test, consistent with the presence of dead zones. If a 5-min experiment was done it is possible the flow would be less evenly distributed.

The free drain and liquid holding volume experiments showed that the amount of water on top, or the difference in head across the column, impacted the velocity of water through the column. With no water on top of the column, no water exited the column.

5.3 Comparison of Intrinsic Permeability and Mean Residence Time Experiments

A sealed double ring infiltrometer (SDRI) test could not be performed to find the exact permeability because the samples could not be fully saturated. If manure samples could be fully saturated with air and water, a connection between the permeability values reported in Chapter 3 and the hydraulic studies in Chapter 4 could be made.

Another way to gain this connection is to perform an instantaneous profile experiment. In this experiment, tensiometers are placed at various elevations on the column (Chiu 2000). In the experiment mentioned, the test is intended to determine the suction front to determine the permeability. However, as mentioned previously, the soil was uniform and poorly graded, unlike the manure tested in these experiments. It would be a great challenge to try and understand a uniform suction front across such a variable substrate.

5.4 Future Work

The experiments should be performed with a SDRI and tensiometers to observe the characteristics of the wetting front. There is no proof that the channels in the manure clog while another is broken open. This is simply speculation. Knowledge of how the manure is getting wet and how much suction occurs along the wetting front is crucial to understanding the hydraulic conductivity. When this test is

performed, more traditional soils methods can be implemented, and the actual hydraulic conductivity can be calculated with this knowledge.

Tracer tests should be performed on manure combined with other media (straw or wood chips). The impact of straw and wood chip additions on intrinsic permeability was minimal, but the impact on the hydraulic characteristics is unknown. It is also possible that the addition of bulking agents could improve the methane production of the system as well by adding a more biologically available nutrient source. Both the hydraulic characteristics and the impact on anaerobic digestion should be investigated with additional substrates. Tracers should also be conducted with potassium bromide as opposed to sodium bromide to avoid possible dehydration of the manure columns over time.

Some improvements for future tracer tests on manure columns would be beneficial and produce more reliable data. The discharge manifold clogged frequently. When the system was set to drain in the sample carboy, a flush of leachate would leave the column. This made taking a grab sample difficult. The column had to be set to drain several minutes before a sample could be collected. A more effective drainage plan should be constructed for future experiments. Using a hose does not work well for drainage; it frequently kinks and does not fit well under the raised floor. The effluent tubing also had problems with air locking. To solve these problems, the manifold should be placed closer to the effluent and in the middle of the columns as opposed to on one side. A smaller tube should be used for the effluent in order to fit under the raised floor.

The biological impact on pore spaces should be investigated. The quantity of substrate consumed as well as the intrinsic permeability after the experiment is completed is crucial. The intrinsic permeability was not measured after the experiment was completed because the wet manure sprayed everywhere and an accurate reading could not be obtained. Another form of finding the intrinsic permeability will be necessary.

The free drain and liquid holding volume experiments take less time than the tracer tests and possibly prove a valid point. The data found during these experiments showed that the potential for water to move across the column increases if there is ponded water on top of the column. Further investigations into the impact of the difference in head across the column should be conducted.

Now that water will go through a column of manure, will leachate? This is the final question that needs to be addressed. The first flush of leachate is very viscous and could clog pore space or the sand and gravel in the column. The next step in this research is to investigate how the leachate will impact hydraulics.

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